

(19)



Europäisches Patentamt

European Patent Office

Office européen des brevets



(11)

EP 0 705 365 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:
19.08.1998 Bulletin 1998/34

(51) Int Cl.⁶: **D21H 11/20, D21H 17/06,
A61L 15/16**

(21) Application number: **94919951.7**

(86) International application number:
PCT/SE94/00613

(22) Date of filing: **21.06.1994**

(87) International publication number:
WO 95/00703 (05.01.1995 Gazette 1995/02)

(54) DEFIBERED FLUFF PULP AND METHOD FOR ITS PREPARATION

ZERFASERTE FLAUMPULPE, UND VERFAHREN ZU IHRER HERSTELLUNG

PATE DUVET DEFIBREE ET PROCEDE DE FABRICATION ASSOCIE

(84) Designated Contracting States:
DE FR

(72) Inventor: **NORLANDER, Lelf
S-791 61 Falun (SE)**

(30) Priority: **23.06.1993 SE 9302166**

(74) Representative: **Hynell, Magnus
Hynell Patenttjänst AB,
Patron Carls väg 2
683 40 Hagfors/Uddeholm (SE)**

(43) Date of publication of application:
10.04.1996 Bulletin 1996/15

(73) Proprietor: **STORA KOPPARBERGS
BERGSLAGS AKTIEBOLAG
S-791 80 Falun (SE)**

(56) References cited:
WO-A-88/04704 US-A- 4 888 093

EP 0 705 365 B1

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

Description

TECHNICAL FIELD

5 The present invention relates to a defibrated fluff pulp containing crosslinked cellulose fibres, having good compressibility under the influence of heat and pressure. The invention also relates to an absorptive body which consists, at least in part, of the said fluff pulp, and also to a method for preparing a fluff pulp, which method includes defibration, i.e. individualizing the cellulose fibres with mechanical devices and crosslinking them.

10 STATE OF THE ART

There has for many years been an interest in chemically crosslinked fluff pulp and it has been proposed that this pulp should be used in absorbent hygiene products. This proposal has been based, in particular, on the favourable properties of the crosslinked cellulose, particularly as regards absorption capacity, wet specific volume and absorption rate, which properties are particularly important when producing absorbent hygiene products such as nappies (diapers), sanitary towels, etc.

In US-A-3 224 926 (1965), Bernadin has described the use of formaldehyde for crosslinking cellulose in the dry and defibrated condition. In addition, the use of other crosslinking reagents has been proposed by, inter alia, Schoggen et al., US 879 678 (1986) (bifunctional aldehydes); Herron, Cooper, US 432 648 (1989) (polyfunctional carboxylic acids); and Norlander, International Patent Application PCT/SE93/00086 (derivatives of dihydroxyethyleneurea). Defibrated cellulose, i.e. individualized cellulose fibres, for example fluff pulp, which is produced in accordance with these methods exhibits a consistent improvement in absorption capacity, wet specific volume and absorption rate.

In addition, PA Graef, WO 88/04704, has reported that a cellulose product produced by treating cellulose with polyfunctional aldehydes, as cellulose crosslinking agents, in the presence of ethylene glycols, propylene glycols or polyether glycols possesses improved absorption properties, principally absorption rate. The crosslinking reaction, if such a reaction takes place, is carried out in a conventional drying machine with the cellulose fibres being in sheet-form, i.e. in the presence of fibre-to-fibre bonds and at temperatures which must not exceed 100°C. The dry matter content associated with any crosslinking reaction which is carried out is stated to be 1-20 %, i.e. the fibres are partially swollen by water. Sheet-fibrating is carried out in subsequent stages in a conventional manner in the dry state prior to shaping the absorptive bodies, which are designed to be used in babies' nappies, feminine products, etc. The increase in absorption capacity as compared with completely untreated fibres is reported to amount to approximately 15 %.

A cellulose product having exposed, dry-crosslinked fibres is normally characterized by a high degree of resilience in the dry state, signifying that high pressures and high temperatures are required in order to compress the product to high densities, for example greater than 0.20-1.0 g/cm³. According to Sultze, PCT/US92/01668, pressures of between 800 and 115,000 psi (55-7930 bar) and temperatures of between 60°C and 180°C are needed in order to achieve this. For several reasons, the high degree of resilience represents a disadvantage. For instance, powerful, and consequently expensive, tools are required for compressing to high density.

BRIEF DISCLOSURE OF THE INVENTION

40 The object of the present invention is to make available a defibrated fluff pulp of the type specified in the preamble, which product possesses a fibre structure having an improved, preferably controllable, compressibility. More particularly, the object of the invention is to make available a fluff pulp for use in absorbent products, more particularly in absorbing pads designed to collect body fluids, for example those products which include babies' nappies, sanitary towels and incontinence products. An especial object in this context is to be able to make available absorbent products which are thinner and more comfortable - but which still possess good absorption properties - than has previously been possible.

A further object is to be able, by means of the improved compressibility, to utilize transport-vehicle and storage-space capacity more efficiently and to decrease transport and storage costs.

50 These and other object can be achieved by the defibrated cellulose product according to the invention therein that the fluff pulp has a fibre structure obtainable by (i) impregnating the fibres with a crosslinking agent and at least one bifunctional, trifunctional or polyfunctional alcohol, (ii) drying the fibres, (iii) defibrating the pulp, and (iv) crosslinking the fibres of the defibrated pulp at a temperature of between 100 and 210°C. By carrying out the defibrating before the crosslinking, the fibres can be individualized more efficiently than would be the case if the crosslinking were to be carried out before the defibration, i.e. good fluffing can be achieved.

55 The cellulose fibres are preferably crosslinked at a temperature greater than 120°C, expediently between 140 and 210°C. Where appropriate, impregnating agents other than bifunctional, trifunctional or polyfunctional alcohols can also provide the effect in accordance with the invention, which effect is expressed by the product gaining a fibre structure

having improved compressibility under the influence of heat and pressure. In this case, the use of such chemicals is also included in the invention. The said crosslinked cellulose fibres probably consist of swollen, and thereby softened, cellulose polymers, which can be obtained by impregnating the cellulose fibres with an effective quantity of bifunctional, trifunctional or polyfunctional alcohols or with some other preparation which causes the fibres to swell.

Another way of characterizing the product according to the invention is that, after having been compressed, it has a density which is at least 15 % higher, under the same conditions as regards compression time, heat and pressure, than that of a cellulose product whose fibres have not been impregnated with bifunctional, trifunctional or polyfunctional alcohols or with any other preparation which causes the fibres to swell, but whose fibres are otherwise of the same type, and are crosslinked with the same amount and agent, as the fibres in the cellulose product according to the invention.

The compressibility of the defibrated and crosslinked fluff pulp, is affected by adding a bifunctional, trifunctional or polyfunctional alcohol expediently together with the crosslinking reagent. The alcohols which are most suitable for use are those which have boiling points (at atmospheric pressure) which exceed the reaction temperature of the crosslinking reaction, i.e. alcohols having boiling points exceeding 100°C, preferably 120°C and expediently exceeding 140°C.

A working hypothesis, formulated in this instance, regarding a probable chemical mechanism involves the hydroxyl groups of the polyfunctional alcohols reacting with the crosslinking reagent in the same way as do the hydroxyl groups of the cellulose. In this way, the alcohols are bound by covalent bonds to the cellulose molecule and the state of swelling of the latter is altered, in association with which the resilience is decreased in the dry state. The addition of alcohol appears to result in a form of softening of the crosslinked cellulose polymer, at least in the dry state and especially at high temperatures. The cellulose fibres which have been chemically crosslinked in this way can be compressed to a substantially higher density, at greatly reduced pressure, as compared with dry-crosslinked cellulose fibres to which none of the said alcohols has been added.

It should be pointed out in this connection that the said hypothesis is not intended to be of any delimiting significance for the scope and validity of the sought-after patent.

Chemicals other than the said alcohols which can cause the cellulose structure to swell can thus also be considered to fall within the scope of the concept of the invention.

Regardless of the correctness of the abovementioned working hypothesis, this invention makes available a method for preparing a fluff pulp, which method is characterized in that the cellulose fibres are impregnated with an effective quantity of chemicals, comprising crosslinking agent and at least one bifunctional, trifunctional or polyfunctional alcohol, in that the impregnated substance is dried, in that it is defibrated, at the latest after drying, and in that the defibrated cellulose fibres are crosslinked in the dry state at a temperature of between 100 and 210°C as a result of which a fluff pulp is obtained having good compressibility under the influence of heat and pressure in combination with good absorption properties.

In this way, cellulose fibre structures are formed, at least in the presence of a high level of one of the said alcohols in the fibres, which can be compressed to the desired density at moderate pressures and temperatures, and which at the same time possess essentially unchanged absorption properties, as compared with crosslinked, conventionally prepared, fluff which has been compressed to the same density but using substantially higher pressure and temperature.

The fluff according to the present invention has a consistently higher absorption capacity and wet specific volume than does a corresponding pulp consisting of untreated, non-crosslinked cellulose fibres compared at the same initial dry density prior to wetting with liquid.

The ability to control the compressibility of a fluff pulp containing crosslinked cellulose fibres is very important when producing absorbent products, such as nappies, sanitary towels, incontinence products, etc. The obvious advantage of the defibrated, crosslinked cellulose product according to the invention is that it can be compressed to high densities at moderate pressures and temperatures but nevertheless be given absorption properties which are equivalent to, or greater than, the corresponding properties of fibre structures which have been formed using non-crosslinked fibre having a substantially lower dry density.

For optimal use of the cellulose fibre, products according to the invention having density gradients, should probably be produced, for example by webs being formed and compressed to different densities prior to assembly, or by pulp having different compressibilities, for example as a result of varying the added quantity of bifunctional, trifunctional or polyfunctional alcohols or equivalent chemicals for impregnating the cellulose fibres, being formed into air-laid or wet-laid webs and then compressed in a common press nip. In this way, an absorptive body can be formed having a density gradient. The number of layers which are used in the absorbent product, and the densities of the different layers, can be determined by the intended area of application. An upper layer of low density can be suitable for products which are to be loaded with large quantities of liquid over a short period of time, which upper layer can face the wearer of the absorbent product, while the layer(s) connected to it can be compressed to (a) higher density (densities).

A baby's nappy of the modern type is nowadays packed at an average density of 130-170 kg/m³. The pulp according to the invention provides excellent opportunities for increasing the average density of the absorptive body in absorbent

products and thereby lowering the costs for transporting and storing these products.

The pulp according to the present invention can also be formed into fibre webs having a high density. The fibre webs can be formed using either a dry or a wet forming technique, with the fibres being dispersed in air or water in conjunction with the web being formed on an endless wire. These webs can be rolled into rolls having a high density, thereby substantially reducing the cost of transporting and storing the pulp as a semi-finished product. The fibre web can subsequently be defibrated, for example in a hammer mill, after which it can be formed into products having the desired density, which density can be substantially lower than the original density of the pulp and entirely dependent on the demands placed on the absorption properties of the final product. Alternatively, the web can be cut into pieces having the desired dimensions and placed directly in the desired position in the absorbent product. A very great advantage in this connection is that the pulp containing the dry-crosslinked cellulose fibres according to the invention can be used in the type of equipment which is nowadays available for producing nappies (also incontinence pads) as well as in machines for manufacturing sanitary towel and air-formed paper.

Webs which are formed out of a pulp according to the present invention can also, on being shaped, be provided with superabsorbent polymer (SAP) in the form of powder or fibres. In this instance, SAP are defined as polymers which can form gels containing at least 10_g of water per g of polymer. Certain applications might require strengthening of the fluff with the aid of long fibres which increase the tear strength. Examples of fibres having this effect are viscose fibres and polyester fibres, but other polymeric fibres can also be used. In thus way, webs can be formed which are sufficiently strong to be incorporated in processes in which webs having a special density can be cut to size and placed in the desired position in absorbent products. Increased strength is also desirable for products having low surface weights, in order to ensure that the absorbing pad does not rupture in the final product.

Such strengthening fibres can also be of interest when manufacturing pulp webs which it is intended to defibrate in conventional defibrating equipment, for example in a hammer mill, for use at a density which is lower than the density of the original web. The reinforcing fibres then contribute to the creation of a fibre structure having a low density and good absorption properties together with substantially increased strength.

A bifunctional trifunctional or polyfunctional alcohol having a molecular weight which exceeds 60_g/mol is expediently used for impregnating the fibres. Where appropriate, the carbon skeletons of the said alcohols can also contain heteroatoms, for example oxygen or nitrogen. The polyfunctional alcohols, or their equivalents, can also, where appropriate, contain one or two polar functional groups of the aldehyde, keto or carboxyl type.

In accordance with the invention, cellulose fibres are expediently impregnated with 2-150 g of bifunctional, trifunctional or polyfunctional alcohol/kg of cellulose fibres. The alcohol preferably consists of glycerol, which is supplied at 5-50 g/kg of cellulose fibre. On the one hand, glycerol is relatively cheap and, on the other, it does not have any toxic or sensitizing properties. Cellulose fibres for crosslinking can be selected from the bleached, partially bleached or unbleached, sulphate-delignified or sulphite-delignified, softwood or hardwood fibre groups. The cellulose fibre can also be selected from the thermomechanical and chemothermomechanical pulp groups.

The following process can be used when producing the dry-crosslinked and compressible fluff pulp. The cellulose fibres are impregnated with aqueous solutions containing the chemicals which are to be added. In this context, crosslinking agents are preferably used which are selected from one of the groups: bifunctional trifunctional or polyfunctional organic acids, bifunctional trifunctional or polyfunctional aldehydes, derivatives of dihydroxyethyleneurea or derivatives of dimethyloldihydroxyethyleneurea. The quantity of crosslinking agent which is added to the cellulose fibres are adjusted within the interval 5-150_g of crosslinking agent/kg of cellulose fibres. The quantity of crosslinking agent is preferably adjusted to the interval 15-50 g of crosslinking agent/kg of cellulose fibre. The subsequent crosslinking can be catalyzed by so-called Lewis acids, for example iron(III) chloride, zinc(II) chloride or magnesium(II) chloride. In addition, substances selected from the groups: alkali metal hypophosphite, alkali metal polyphosphate, alkali metal phosphate, alkali metal sulphate, sodium fluoroborate, disodium carbonate and organic amines can catalyze all the abovementioned crosslinking reagents. The above-mentioned bifunctional trifunctional or polyfunctional alcohols, or their equivalents, are also added to the said aqueous solution.

The cellulose fibre is then formed into a web which is dried at temperatures which are sufficiently low to ensure that the crosslinking reagent is not activated prior to the subsequent defibration which then takes place in the dry state, i.e. at a dry matter content which exceeds 80 %, and is preferably between 90 and 95 %. The dry defibration is expediently carried out using a hammer mill. Alternatively, the cellulose fibres are defibrated before the drying process, thus in the wet state, resulting in the cellulose fibre being twisted during drying, something which is well known from the manufacture of so-called flash-dried pulp. Defibration in the wet state is carried out at a dry matter content of 30-80 %, preferably at 40-55 %.

The defibrated and dried cellulose fibres are crosslinked, in the dry and individualized state, by the crosslinking reagent which has been added and being activated by means of heating to reaction temperature. The heating of the material preferably takes place by means of the individualized cellulose fibre being dispersed in hot air. The reaction temperatures which are required depend on the crosslinking reagent employed. In order to achieve technically acceptable reaction times, the reaction is expediently carried out at 140-190°C. The crosslinking reaction is then carried out

using a dwell time of between 1 and 30 minutes, preferably between 2 and 10 minutes.

Further characteristics and aspects, and also advantages, of the invention are evident from the subsequent patent claims and from the following account of experiments which have been carried out, and from the subsequent description of a preferred embodiment of the method according to the invention.

BRIEF DESCRIPTION OF THE FIGURES

In the following description of experiments which have been carried out, reference will be made to the attached drawings, of which

- Fig. 1 shows how absorption properties and specific volume are determined in accordance with Scan-C 33:80,
- Fig. 2 illustrates a method for determining absorption rate (acquisition),
- Fig. 3 shows the absorption capacity as a function of the initial density in the dry state when making measurements on compressed fluff pulp which has been crosslinked in the dry state,
- Fig. 4 shows the density as a function of the applied sheet pressure at 90°C when compressing fluff pulp which has been crosslinked in the dry state,
- Fig. 5 shows the wet specific volume as a function of the initial density for compressed fluff pulp which has been crosslinked in the dry state,
- Fig. 6 shows, in the form of a diagram, the absorption rate as a function of the thickness of the test sample, firstly in a cellulose product according to the invention which has been supplied with superabsorbent powders (SAP), and secondly in a reference material which also contains superabsorbent of the same type,
- Fig. 7 constitutes a flow diagram which schematically illustrates a first preferred embodiment of the method for preparing fluff pulp, or other defibrated cellulose product, according to the invention, and
- Fig. 8 constitutes a flow diagram which schematically illustrates a second preferred embodiment of the method for preparing fluff pulp, or other defibrated cellulose product, according to the invention.

DESCRIPTION OF MEASUREMENT METHODS

Determination of specific volume and absorption properties in accordance with modified Scan-C 33:80

Definitions

Absorption capacity:

The ratio between the mass (weight) of the liquid which is taken up by a standard test sample of the fluff under defined conditions and the original mass (weight) of the test sample.

Specific volume:

Volume of the test piece in the dry state (dry specific volume) or in the wet state (wet specific volume) per unit mass (unit weight) of fluff.

For the measurements, test pieces of fluff are used which have a mass of 1.0 g and a diameter of 50 mm and which have been formed, by air forming, in pad form of the same type as is described in Scan-C 33:80. The density of the test pieces after forming is between 45 and 60 kg/m³.

The test pieces were compressed at different pressures and temperatures in a laboratory press having thermostated press plates. The thickness of the test pieces was determined using a spring-loaded thickness gauge which loads the sample with 2.5 kPa on a circular surface area of 3.14 cm². The thickness of the compressed test pieces was determined at least one hour after compressing, following conditioning at 23°C and 50 % RH.

An apparatus of the type which is shown in Fig. 1 was used for the measurements. The test piece 1 is placed vertically and a weight 2 is placed on top of it, which weight loads the test pieces with a pressure of 2.5 kPa. The test piece is permitted to absorb water from below through a perforated bed 3. The water level is adjusted such that the underside of the test sample only just dips down into the water. The test sample is allowed to absorb water for 30 seconds, after which the water level is lowered. The test piece is then allowed to drain for 30 seconds, after which the weight 2 is removed and the wet test piece is weighed.

After weighing, the thickness of the test piece in the wet state is determined using a spring-loaded thickness gauge which loads the sample with 2.5 kPa on a circular surface area of 3.14 cm². The reading is taken 30 seconds after the load has been applied.

The absorption capacity is calculated from the expression $Y = (b-w)/w$, where

EP 0 705 365 B1

Y = the absorption capacity in g/g

b = the mass of the wet test sample in g

The wet specific volume is calculated from the expression $X = A \times h \times w$, where

X = specific volume in dm³/kg

A = the surface area of the bottom of the test sample in cm²

w = the mass of the dry test sample in g

h = the height in cm of the test sample while being loaded in the dry state and the wet state, respectively.

The dry density is calculated from

$$D = \frac{w}{A \times h} \times 1000 \text{ (kg/m}^3\text{)}$$

Determination of absorption rate (acquisition)

An apparatus which is shown schematically in Fig. 2 was used for these measurements. The sample 10 is covered with a non-woven, 17 g/m² spun-bonded polypropylene from Fibreweb and placed under a plate 11, which was loaded with weights 12 so that the sample 10 was subjected to a compression pressure of 2.6 kPa. The plate 12 has the same dimensions, 12*30 cm, as the sample 10. There is a circular hole in the rectangular plate and the hole is connected to a pipe 13 which has the same diameter, 50 mm, as the said hole. A metering hose has been designated 14; a pair of electrodes at the bottom of the pipe 13, immediately above the sample 10, have been designated 15, 16; and electronic and chronometric equipment has been designated 17.

Per litre, the synthetic urine contained: 9.0 g of NaCl, 2.69 g of KH₂PO₄, approximately 1.8 g of Na₂HPO₄ and 0.1 g of acid fuchsin, with the remainder essentially being distilled water. The pH of the liquid was 6.1-6.4.

50 ml of the liquid was metered down into the pipe 13 in 5 s from the metering hose 14. The liquid in the pipe has been designated 18. The time was measured which was taken for the sample to absorb and spread the liquid 18, i.e. the time it took to empty the pipe 13 of all liquid. Each test was carried out 4 times, i.e. 4 doses of synthetic urine were applied to the sample at 5 min intervals, with each dose consisting of 50 ml of synthetic urine.

EXPERIMENTAL SERIES 1

A4 sheets of fluff pulp of the bleached softwood sulphate type (STORA Fluff EC 0.1) were conditioned at 23°C and 50 % RH (relative humidity), and weighed. The sheets were impregnated with chemicals by dipping, for 15 s, into solutions having the composition given in Table 1.

The wet sheets were pressed, between absorbent paper, in a laboratory press to a dry matter content of a good 50 %, at a sheet pressure of approximately 5 bar. The sheets were dried at 80°C for 2 hours while being clamped under a dryer felt on a drying cylinder, after which drying of the sheets was completed by heating at 110°C for 25 min. The dried sheets were weighed and the amount of chemicals applied (amount added per amount of cellulose) was calculated.

The impregnated sheets were conditioned at 50 % RH and 23°C for 4 hours, and were then defibrated in a Kamas HO1 hammer mill at 4500 revolutions/min, so that the fibres were substantially individualized. The defibrated material was heated up to the reaction temperature in a warm-air oven in which thermostatted air is blown through a bed of the material. The defibrated material was kept in the oven at the reaction temperature for 6 min.

EP 0 705 365 B1

Table 1

| Sample No. | Crosslinking agent citric acid g/dm ³ | Catalyst disodium hydrogen phosphate g/dm ³ | Impregnating agent alcohol | | | | Total amount applied g/kg |
|--------------------|--|--|----------------------------|-----------------------|-----------------------|-----------------------|---------------------------|
| | | | A1) g/dm ³ | B2) g/dm ³ | C3) g/dm ³ | D4) g/dm ³ | |
| 1:0 | 50 | 19 | - | - | - | - | 62 |
| 1:2 | 50 | 19 | 19 | - | - | - | 79 |
| 1:3 | 50 | 19 | 63 | - | - | - | 115 |
| 1:4 | 50 | 19 | - | 63 | - | - | 114 |
| 1:5 | 50 | 19 | - | - | 63 | - | 120 |
| 1:6 | 50 | 19 | - | - | - | 63 | 123 |
| 1:10 ⁵⁾ | | | | | | | 0 |

1) Alcohol A = glycerol

2) Alcohol B = diethylene glycol

3) Alcohol C = triethylene glycol

4) Alcohol D = 2-hydroxymethyl-2-methylpropanediol

5) 1:10 = reference, ie. not crosslinked and not treated with alcohol

Table 2

| Sample No. | Temperature °C | Diameter cm | Sheet pressure kPa $\times 10^{-2}$ | Density kg/m ³ at 2.5 kPa | Spec.vol. wet Dm ³ /kg at 2.5 kPa | Absorption capacity g/g | Relative density % |
|------------|----------------|-------------|-------------------------------------|--------------------------------------|--|-------------------------|--------------------|
| 1:0:A | 90 | 5.40 | 6 | 96 | 10.07 | 9.80 | 100 |
| 1:0:B | 90 | 5.40 | 12 | 110 | 9.66 | 9.52 | 100 |
| 1:0:C | 90 | 5.45 | 26 | 157 | 8.72 | 8.38 | 100 |
| 1:0:D | 90 | 5.50 | 50 | 182 | 8.55 | 8.14 | 100 |
| 1:0:E | 130 | 5.50 | 50 | 285 | 8.03 | 7.60 | 100 |
| 1:2:A | 90 | 5.40 | 6 | 123 | 9.22 | 9.07 | 128 |
| 1:2:B | 90 | 5.45 | 12 | 140 | 8.93 | 8.69 | 128 |
| 1:2:C | 90 | 5.45 | 26 | 219 | 7.83 | 7.50 | 140 |
| 1:2:D | 90 | 5.50 | 50 | 224 | 8.50 | 8.10 | 123 |
| 1:2:E | 130 | 5.50 | 50 | 354 | 7.65 | 7.30 | 124 |
| 1:3:A | 90 | 5.50 | 6 | 132 | 8.64 | 8.37 | 137 |
| 1:3:B | 90 | 5.50 | 12 | 200 | 8.24 | 7.80 | 184 |
| 1:3:C | 90 | 5.40 | 26 | 230 | 7.69 | 7.45 | 147 |
| 1:3:D | 90 | 5.50 | 50 | 351 | 7.43 | 7.48 | 193 |
| 1:3:E | 130 | 5.45 | 50 | 471 | 6.92 | 6.70 | 166 |
| 1:4:A | 90 | 5.40 | 6 | 104 | 9.52 | 9.28 | 108 |
| 1:4:B | 90 | 5.40 | 12 | 130 | 8.74 | 8.75 | 118 |
| 1:4:C | 90 | 5.45 | 26 | 146 | 8.16 | 7.82 | 93 |
| 1:4:D | 90 | 5.50 | 50 | 254 | 8.36 | 7.89 | 139 |

Table 2 (continued)

| Sample No. | Temperature °C | Diameter cm | Sheet pressure kPa $\times 10^{-2}$ | Density kg/m ³ at 2.5 kPa | Spec.vol. wet Dm ³ /kg at 2.5 kPa | Absorption capacity g/g | Relative density % |
|------------|----------------|-------------|-------------------------------------|--------------------------------------|--|-------------------------|--------------------|
| 1:4:E | 130 | 5.50 | 50 | 340 | 7.86 | 7.00 | 119 |
| 1:5:A | 90 | 5.40 | 6 | 101 | 9.80 | 9.23 | 105 |
| 1:5:B | 90 | 5.50 | 12 | 124 | 9.02 | 8.76 | 113 |
| 1:5:C | 90 | 5.45 | 26 | 148 | 8.53 | 8.32 | 95 |
| 1:5:D | 90 | 5.50 | 50 | 185 | 8.50 | 7.98 | 101 |
| 1:5:E | 130 | 5.55 | 50 | 328 | 7.35 | 6.80 | 115 |
| 1:6:A | 90 | 5.40 | 6 | 176 | 8.17 | 7.61 | 183 |
| 1:6:B | 90 | 5.45 | 12 | 223 | 7.74 | 7.19 | 204 |
| 1:6:C | 90 | 5.50 | 25 | 270 | 7.31 | 7.04 | 172 |
| 1:6:D | 90 | 5.50 | 50 | 373 | 7.31 | 6.99 | 204 |
| 1:6:E | 130 | 5.50 | 50 | 463 | 6.65 | 6.30 | 163 |
| 1:10:A | 20 | 5.50 | 6 | 150 | 7.69 | 7.60 | |
| 1:10:B | 20 | 5.50 | 12 | 217 | 7.12 | 6.50 | |
| 1:10:C | 20 | 5.40 | 26 | 337 | 6.25 | 5.80 | |
| 1:10:D | 20 | 5.40 | 52 | 453 | 5.51 | 5.00 | |

Test pieces having a weight of 1 g and a diameter of 50 mm were formed in accordance with the above description. The test pieces were then compressed at different pressures and temperatures in a laboratory press using heated press plates. The true sheet pressure and resulting density were calculated after measuring the diameter and thickness of the compressed test piece.

The results which were obtained are shown in Table 2. The results in Table 2 have been illustrated in diagrammatic form in Figs. 3-5. In these figures, the curves have been drawn using the corresponding data in Table 2. In addition, the concept of relative density has been introduced. The definition of this is as follows: pair-wise comparison of crosslinked pulp with pulp which was crosslinked in the presence of polyfunctional alcohol containing a corresponding quantity of crosslinking agent. The compression was carried out using the same conditions as regards pressure, temperature and time. The density of the pulp which was only crosslinked is then given the value of 100 %.

Fig. 3 shows the absorption capacity as a function of the density in the dry state when carrying out measurements on dry-crosslinked, compressed fluff pulp. It is evident from this figure that the completely untreated fluff pulp (1:10, reference which was not crosslinked and not treated with alcohol) has the worst absorption capacity at all initial dry densities. It is also evident that the fluff pulp which was only crosslinked (1:0, which was not treated with alcohol) has an absorption capacity which is only marginally better than that of fluff pulp which was treated with alcohol in accordance with this invention (1:2, 1:3, 1:4, 1:5 and 1:6).

Fig. 4 shows the density as a function of the applied sheet pressure when compressing, at 90°C, fluff pulp which was crosslinked in the dry state. The completely untreated fluff pulp (1:10) is most readily compressed, due to the fact that it is not crosslinked. Nevertheless, the crosslinked fluff pulp which was treated with triethylene glycol (1:5) has compression properties which are largely unchanged, showing that the chemical structure of the alcohols also has an effect on the result.

Fig. 5 shows the wet specific volume as a function of the initial density in compressed fluff pulp which was crosslinked in the dry state. In this comparison, the completely untreated fluff pulp (1:10) cannot hold as much liquid as can the other fluff pulp samples. High wet specific volumes at high densities are found for the other fluff pulp samples which were treated with alcohol (1:2, 1:3, 1:4, 1:5 and 1:6). This is especially evident in samples 1:3 and 1:6 which, along the whole of the respective curves, have a wet specific volume which is equivalent to that of the untreated fluff pulp sample, at a density which is at least 50 kg/m³ higher.

EXPERIMENTAL SERIES 2

The function of the alcohols in decreasing resilience in the dry state has also been tested when using other crosslinking agents, such as a methyl derivative of dimethyldihydroxyethyleneurea, DMDHEU (commercial product from Société Française Hoechst and having the trade name Arkofix NZF), as is evident from Tables 3 and 4. Catalyst V3881 consists of an acidic solution (pH = 0) of magnesium chloride and acetic acid. The concentrations which are given with regard to Arkofix NZF and catalyst V3881 relate to absolutely dry product after determining dry matter content in an incubator at 110°C. The procedure for carrying out Experimental series 2 is similar to the above described procedure in all respects apart from the fact that the reaction conditions for crosslinking with the said methyl derivative of dimethyldihydroxyethyleneurea (Arkofix NZF) are that the crosslinking takes place at 160°C for 3 min.

Table 3

| Sample No. | Crosslinking agent Arkofix NZF* g/dm ³ | Catalyst V3881 g/dm ³ | Impregnating agent glycerol g/dm ³ | Amount applied g/kg |
|------------|--|----------------------------------|--|---------------------|
| 2:1 | 59 | 2 | - | 50 |
| 2:2 | 59 | 2 | 59 | 99 |

*Trade name for methyl derivative of dimethyldihydroxy-ethyleneurea, DMDHEU

Table 4

| Sample No. | Temperature °C | Diameter cm | Sheet pressure kPa $\times 10^{-2}$ | Density kg/m ³ at 2.5 kPa | Spec. vol. wet dm ³ /kg at 2.5 kPa | Absorption capacity g/g | Relative density % |
|------------|----------------|-------------|-------------------------------------|--------------------------------------|---|-------------------------|--------------------|
| 2:1:A | 90 | 5.50 | 25 | 156 | 8.41 | 7.9 | 100 |
| 2:1:B | 90 | 5.45 | 50 | 216 | 7.65 | 7.2 | 100 |
| 2:2:A | 90 | 5.45 | 25 | 367 | 6.67 | 6.0 | 235 |
| 2:2:B | 90 | 5.40 | 50 | 465 | 6.23 | 5.9 | 216 |

EXPERIMENTAL SERIES 3

An excess of aqueous solutions having the composition as given in Table 5 (remainder water) was added to a pulp web based on Nordic softwood sulphate pulp. The excess of the solution was pressed out in a single-felted press nip.

Table 5

| Sample No. | Crosslinking agent citric acid g/dm ³ | Catalyst NaH ₂ PO ₄ g/dm ³ | Impregnating agent glycerol g/dm ³ | Dry matter content after pressing % | Calculated amount applied g/kg |
|------------|--|---|---|-------------------------------------|--------------------------------|
| 3:1 | 31 | 12 | - | 49.1 | 45 |
| 3:2 | 31 | 12 | 25 | 49.0 | 71 |
| 3:3 | 31 | 12 | 50 | 49.8 | 94 |
| 3:4 | 38 | 15 | 75 | 51.5 | 121 |
| 3:5 | 48 | 19 | 50 | 51.5 | 110 |
| 3:6 | 48 | 19 | - | 49.6 | 68 |

The moist web of cellulose fibre was torn in a toothed screw into pieces having a size of about 1 centimetre, which pieces were defibrated twice, without previous drying, in a 20-inch disc refiner. Immediately after the cellulose fibre had passed through the disc refiner in the second defibration stage the fibre, which had thus been substantially individualized, was dried by being dispersed in heated air. The cellulose fibre was dried from a dry content of approximately 50 % to a dry content of approximately 90 %. The crosslinking reaction was then initiated by heating the fibre material at 180°C for 6 min.

EP 0 705 365 B1

The resulting cellulose product was then examined in the same way as in Experimental series 1 and 2. The results are given in Table 6.

Table 6

| Sample No. | Temperatur °C | Diameter cm | Sheet pressure kPa $\times 10^{-2}$ | Density kg/m ³ at 2.5 kPa | Spec. vol. wet dm ³ /kg at 2.5 kPa | Absorption capacity g/g. | Relative density %- |
|------------|---------------|-------------|-------------------------------------|--------------------------------------|---|--------------------------|---------------------|
| 3:1:A | 90 | 5.60 | 24 | 175 | 10.41 | 10.0 | 100 |
| 3:1:B | 90 | 5.60 | 48 | 185 | 9.95 | 10.2 | 100 |
| 3:2:A | 90 | 5.55 | 25 | 230 | 9.29 | 9.1 | 131 |
| 3:2:B | 90 | 5.60 | 48 | 339 | 8.76 | 8.6 | 183 |
| 3:3:A | 90 | 5.55 | 25 | 254 | 8.97 | 8.6 | 145 |
| 3:3:B | 90 | 5.55 | 49 | 390 | 8.00 | 7.8 | 211 |
| 3:4:A | 90 | 5.60 | 24 | 336 | 8.25 | 8.1 | 192 |
| 3:4:B | 90 | 5.60 | 48 | 376 | 7.88 | 7.5 | 204 |
| 3:5:A | 90 | 5.60 | 24 | 208 | 9.01 | 8.8 | 188 |
| 3:5:B | 90 | 5.50 | 50 | 348 | 8.10 | 7.9 | 255 |
| 3:6:A | 90 | 5.55 | 25 | 111 | 11.03 | 11.4 | 100 |
| 3:6:B | 90 | 5.55 | 49 | 136 | 10.42 | 10.1 | 100 |

It is evident from the above Tables and the attached Figure drawings 3-5 that the compressibility of a cellulose product which consists essentially of fibres which have been crosslinked in the dry state in the presence of bifunctional, trifunctional or polyfunctional alcohols, and thereby gained the said structure of cellulose fibres, can be controlled by the amount and type of the said bifunctional, trifunctional or polyfunctional alcohols which are added.

When an alcohol is added, the cellulose product according to the invention, and its structure of cellulose fibres, are compressed to a substantially greater extent than is fluff pulp at the same compression pressure when this fluff pulp has only been crosslinked. At comparable initial dry densities, a cellulose fibre structure according to the invention and a cellulose fibre structure which is only crosslinked also have comparable absorption properties. While having a substantially higher dry density prior to wetting, a cellulose fibre structure according to the invention has an absorption capacity/wet specific volume which is as high as that of cellulose fibre structures which have not been crosslinked.

EXPERIMENTAL SERIES 4

Experimental series 4 was designed with the aim of reviewing further the possibilities for using the dry-crosslinked and compressed fibre in absorbent products, such as, for example, babies' nappies. More particularly, the aim of the experiments was to study absorption rate and its dependence on the thickness/degree of compression of the absorptive pad.

Present-day standard nappies are often packed at densities within the range 150-200 kg/m³. However, a certain spring-back takes place during use/evaluation. Reference samples, of which the reference material in sample No. 3, Table 7 below, can, in particular, be regarded as representing the absorptive pad in a baby's nappy of the modern type, as regards performance (absorption rate) and thickness, were included in the experimental series. An essential property of nappies is the rate with which the liquid is absorbed by the absorptive pad. A low rate of absorption results in an increased risk of leakage since the urine which is not absorbed promptly by the absorptive pad must be kept enclosed within the nappy by different forms of sealing functions. The absorption rate in association with the first dosing with liquid is judged not to be critical for current standard nappies. On the other hand, the decreased rate of absorption associated with increasing liquid loading entails an increasing risk of leakage.

A commercial fluff pulp, known under the trade name Stora Cell EC 0.1 (manufacturer, Stora Kopparbergs Bergslags AB), which is a sulphate fluff pulp based on Nordic softwood fibre, was used as the reference material. The pulp was defibrated at 3500 rpm in a Kamas HO1 hammer mill having an 8 mm sieve. Pads were formed with an air-laying technique using the defibrated material. A superabsorbent (SAP) was used which is known under the trade name Drytech 510 (manufacturer Dow Chemical Corp.), which, according to information from the manufacturer, is defined as a crosslinked acrylate copolymer. The superabsorbent, Drytech 510 HC, was added to the pads at different

levels in three additions. The pads were pressed in a press under the conditions of pressure and temperature given in Table 7. When being used for evaluating absorption rate, all the pads had the dimensions 12*30 cm but varied in thickness and density.

The novel fibre which was produced in accordance with Example 3:2, Table 5, was formed into sheets having a grammage of approximately 700 g/m² and having the dimensions 12x30 cm. The sheets were compressed at approximately 20 bar and at 130°C for 30 s, resulting in a sheet density of approximately 400 kg/m³. The material was cut into strips of approximately 4.7 cm in width which were then defibrated at 2500 rpm in a Kamas HO1 laboratory mill having an 8 mm sieve. The fluffed material was subsequently formed into pads with an air-laying technique and superabsorbent, Drytech 510 HC, was distributed in the sheet by being added in three batches. The formed sheets were pressed in a press under the conditions of pressure and temperature given in Table 7. When being used for evaluating absorption rate, all the sheets had the dimensions 12x30 cm but varied in thickness and density.

As can be seen from Table 7 and diagram, an absorptive pad based on a conventional fluff pulp which has not been crosslinked loses, to a large extent, the important function of rapidly absorbing liquid if it has been compressed to high initial dry density or otherwise pressed out into a thin absorptive pad. By contrast, an absorptive pad based on the novel fibre can be compressed to a thickness which is less than half the thickness of, for example, the reference sample 3, Table 7, while having an absorption rate which is largely maintained or, in several cases, even increased, when the test piece has been loaded with liquid, i.e. in dosages 2-4. The results thus demonstrate that the novel fibre provides substantially improved possibilities for developing thin, but nevertheless functionally satisfactory, absorbent products having a high absorption rate, and thus also decreased leakage frequency, as compared with a chemical fluff pulp which contains a high proportion of superabsorbent and is only compressed and not crosslinked.

There are many options for varying the design of the absorptive pad. For example, the densities in different layers, the distribution and type of super-absorbent, the non-uniform grammage profile, etc. can be varied. All these aspects have an influence on absorption rate. The examples should thus not be seen as a limitation of the invention but are only intended to demonstrate differences in performance between a present-day commercial, chemical fluff pulp and the novel pulp when being used in thin, absorbent products, particularly when the absorptive pad has been compacted to a high density, i.e. >200 kg/m³.

Table 7

| | Reference | | Examples of absorptive pads according to the invention | | | | | | | |
|------------------------------------|---------------------------|------------|--|------------|------|------|------|------|------|------|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | | |
| Sample No. | | | | | | | | | | |
| Pulp | | Stora Cell | Stora Cell | Stora Cell | | | | | | |
| | | EC 0.1 | EC 0.1 | EC 0.1 | 3:2* | 3:2* | 3:2* | 3:2* | 3:2* | 3:2* |
| Surface weight value, target value | g/m ² | 1000 | 1000 | 1000 | 1000 | 1000 | 1000 | 1000 | 1000 | 800 |
| Surface weight SAP*, target value | g/m ² | 140 | 140 | 140 | 140 | 140 | 140 | 140 | 140 | 140 |
| Pressing: | | | | | | | | | | |
| Pressure | kPa·10 ⁻² | 18.1 | 10.9 | 7.4 | 14.3 | 8.0 | 5.1 | 14.1 | 8.1 | |
| Time | s | 40 | 20 | 30 | 40 | 15 | 10 | 80 | 12 | |
| Temperature | °C | 20 | 20 | 20 | 90 | 90 | 90 | 130 | 90 | |
| Sample weight incl. SAP | | | | | | | | | | |
| (12*30) | g | 41.6 | 41.8 | 41.9 | 41.6 | 42.2 | 42.0 | 42.5 | 36.3 | |
| Quantity of SAP | g | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | |
| Surface weight, (fluff + SAP) | g/m ² | 1202 | 1206 | 1234 | 1152 | 1164 | 1153 | 1159 | 993 | |
| Thickness (") | mm | 4.0 | 5.9 | 8.0 | 3.0 | 5.0 | 7.0 | 2.2 | 4.1 | |
| Density (") | kg/m ³ | 297 | 205 | 154 | 381 | 235 | 165 | 539 | 242 | |
| Absorption rate - : | 4 x 50 cm ³ /s | | | | | | | | | |
| Dosage 1 | cm ³ /s | 0.91 | 2.32 | 5.75 | 1.85 | 2.44 | 4.68 | 1.37 | 2.18 | |
| Dosage 2 | cm ³ /s | 0.37 | 0.68 | 1.49 | 1.38 | 1.57 | 2.08 | 1.03 | 1.31 | |
| Dosage 3 | cm ³ /s | 0.26 | 0.45 | 0.86 | 1.08 | 1.15 | 1.42 | 0.76 | 0.96 | |
| Dosage 4 | cm ³ /s | <0.18 | 0.33 | 0.59 | 0.76 | 0.89 | 1.15 | 0.50 | 0.67 | |

DESCRIPTION OF INDUSTRIAL EMBODIMENTS

Fig. 7 schematically illustrates a first method of implementing the invention in full scale in continuously operating processes. The upper part of the schematic flow diagram in Fig. 7 illustrates how and where the crosslinking chemicals as well as the alcohols according to this invention can be added to the process, in accordance with one example, and the lower part of the flow diagram shows the subsequent preparation, comprising defibration and crosslinking.

A furnish is pumped through a headbox 30 out onto a wire section 31. A press section has been generally designated 32. The press section comprises a number of presses 33, 34 and a dryer 35. The press section 32 can also contain several presses upstream and downstream of the dryer 35, which can also be termed a pre-dryer.

The crosslinking reagent and alcohols which are used in accordance with the invention, together with catalyst(s), are added, in accordance with the preferred embodiment, to the web-shaped cellulose sheet before the latter is dried, expediently in the press section at a point in the process when the pulp has a dry content amounting to at least 20 %, expediently a dry matter content in the range 30-50 %. Different methods and arrangements are conceivable for ap-

plying the chemicals so that they are efficiently incorporated into the pulp material. For example, the chemical solution can conceivably be sprayed onto the pulp from one or more transverse sprinkler pipes and integration of the chemicals into the pulp subsequently effected using a downstream press 36. Preferably, however, the chemicals are expediently supplied using an apparatus 37 of the type which is conventionally used as a size press, this being a well-tried technique for incorporating chemicals into relatively thick and bulky sheets. The web material, which now contains the chemicals, is dried in the dryer 35 at a temperature below the crosslinking temperature of the chemicals, preferably at a web temperature of less than 100°C.

After the dryer 35, the web-shaped sheet 38 can be reeled up into roller bales 39 or - alternatively - conducted directly to a plant for defibering and hardening. This unit has been designated 40 in Fig. 7. The web material 38 is next conducted into a defibrator 41. An apparatus which is suitable for dry defibration is a hammer mill, which is available in different designs. Other equipment, for example disc refiners, are also conceivable in principle for bringing about dry defibration, but a hammer mill is to be preferred in relation to dry defibration.

As a result of being defibrated in the apparatus 41, the material is fluffed and consequently achieves a high specific volume. At the same time, it also loses most of its fibre-binding properties and is therefore difficult to re-shape into an interconnected web. However, it is desirable to be able to carry out the crosslinking, which follows the defibration, under continuous conditions, which permit regulation of the crosslinking time and crosslinking temperature, and which simultaneously ensure that the pulp is treated uniformly in all parts of the bulky, fluffed material. According to a preferred embodiment, the fluffed pulp 42 is fed in between two endless, relatively wide-apart, wires 43, 44. The fluffed pulp 42 is, as it were, confined in between the wires 43, 44 and is fed slowly through a tunnel oven 45. Warm gas flows through the oven 45. By means of adjusting the feeding speed of the wires 43, 44, the dwell time of the pulp 42 in the oven 45 at reaction temperature is expediently regulated to be between 1 and 30 min, preferably to be between 2-10 min. The temperature in the oven is regulated to be between 100 and 210°C, preferably to be greater than 120°C, expediently to be between 140 and 190°C, in association with which the added chemicals are activated and initiate the intended reactions with the cellulose molecules. After treatment in the oven 45, the pulp is expediently cooled, which can be carried out in a separate cooling zone.

The material which has been prepared in this way is then pressed together once again in a press 46 to form a more or less interconnected material 47, which is folded and baled in a baling press 48. The finished bales have been designated 49. Alternatively, the material 47 can be conveyed directly to a nappy manufacturing plant 50 or to a plant for manufacturing other absorbent products. This plant can in principle be connected directly after the oven 45, thereby eliminating the need for additional fluffing. As a further alternative, the material can be rolled into rolls (not shown), as has been mentioned above.

Fig. 8 schematically illustrates a second method of implementing the invention in full scale in continuously operating processes. Roll or bale pulp 60 is conveyed to a disintegrator, a so-called pulper 61, where the pulp 60 is dispersed in water 62, which contains the relevant chemicals. The slurry which is obtained in this way is dewatered. This can be effected, for example, in a thickener, designated generally by 63. In the example, the thickener contains, in a conventional manner, a suction cylinder wire 64 and a press section 65. A pre-dryer may also be present. The dewatering gives rise to a so-called wet pulp which has a dry content of 45-50 %. The wet pulp is defibrated in disc refiners 66, 67 and is then dried in a flash dryer 68 at a fibre temperature of less than 100°C and is finally heated in a further tower 69 in order to be thoroughly heated homogeneously immediately before the activation of the crosslinking chemicals, which can be carried out in the manner which has been described in association with the preceding examples. This part of the process, comprising treatment in the units 42-50, will not therefore be described in any further detail here, and reference will simply be made to the preceding description.

Claims

1. Defibrated fluff pulp containing crosslinked cellulose fibres, having good compressibility under the influence of heat and pressure, **characterized** in that the fluff pulp has a fibre structure obtainable by (i) impregnating the fibres with a crosslinking agent and at least one bifunctional, trifunctional or polyfunctional alcohol, (ii) drying the fibres, (iii) defibrating the pulp, and (iv) crosslinking the fibres of the defibrated pulp at a temperature of between 100 and 210°C.
2. Fluff pulp according to claim 1, **characterized** in that the cellulose fibres have been crosslinked at a temperature greater than 120°C.
3. Fluff pulp according to claim 2, **characterized** in that the cellulose fibres have been crosslinked at a temperature of between 140 and 190°C.

4. Fluff pulp according to any one of claims 1-3 **characterized** in that the crosslinked cellulose fibres consist of swollen, and thereby softened, cellulose polymers.
- 5 5. Fluff pulp according to any one of claims 1-4, **characterized** in that, after having been compressed, it has a density which is at least 15% higher, under the same conditions as regards compression time, heat and pressure, than that of a cellulose product whose fibres have not been impregnated with a bifunctional, trifunctional or polyfunctional alcohol, or with some other preparation which provides an equivalent effect, but which fibres are otherwise of the same type, and are crosslinked with the same agent, as the fibres in the first-named fluff pulp.
- 10 6. Fluff pulp according to any one of claims 1-5, **characterized** in that the said bifunctional, trifunctional or polyfunctional alcohols have a molecular weight which exceeds 60 g/mole.
- 15 7. Fluff pulp according to any one of claims 1-6, **characterized** in that the carbon structure of the said bifunctional, trifunctional or polyfunctional alcohols contain one or more heteroatoms, which heteroatoms can comprise any one of oxygen or nitrogen.
- 20 8. Fluff pulp according to any one of claims 1-7, **characterized** in that the said bifunctional, trifunctional or polyfunctional alcohols contain one or two polar functional groups of the type of groups which comprise aldehyde groups, keto groups and carboxyl groups.
- 25 9. Fluff pulp according to any one of claims 1-8, **characterized** in that the cellulose fibres are impregnated with 2-150 g of a bifunctional, trifunctional or polyfunctional alcohol/kg of cellulose fibres.
- 30 10. Fluff pulp according to any one of claims 1-9, **characterized** in that the cellulose fibres are impregnated with 5-50 g of a bifunctional, trifunctional or polyfunctional alcohol/kg of cellulose fibres.
- 35 11. Fluff pulp according to any one of claims 1-10, **characterized** in that it possesses a fibre structure having an improved compressibility under the influence of heat and pressure, obtainable by impregnating the cellulose fibre with an effective quantity of glycerol.
- 40 12. Fluff pulp according to any one of claims 1-11, **characterized** in that, when an air-laid absorptive pad produced from the cellulose product is compressed to a dry density which is at least 50 kg/m³ higher than that of an air-laid absorptive pad produced from untreated fibres, it has a wet specific volume which at least essentially corresponds to that of the untreated, compressed product, i.e. a cellulose product essentially consisting of cellulose fibres which have neither been crosslinked nor impregnated with bifunctional, trifunctional or polyfunctional alcohols or with any other equivalent reagent.
- 45 13. Fluff pulp according to any one of claims 1-12, **characterized** in that, when an air-laid absorptive pad produced from the cellulose product is compressed to a dry density which is at least 50 kg/m³ higher than that of an air-laid absorptive pad produced from untreated fibres, it has an absorption capacity which is at least as great as or greater than that of the compressed product produced from the untreated cellulose pulp, i.e. a cellulose product essentially consisting of cellulose fibres which have neither been crosslinked nor impregnated with bifunctional, trifunctional or polyfunctional alcohols or with any other equivalent reagent, which absorptive pad produced from untreated cellulose fibres has been compressed to 200 kg/m³.
- 50 14. Fluff pulp according to any one of claims 1-13, **characterized** in that it contains 10-150 g of crosslinking agent/kg of cellulose fibres.
- 55 15. Fluff pulp according to any one of claims 1-14, **characterized** in that it contains 20-60 g of crosslinking agent/kg of cellulose fibres.
16. Fluff pulp according to any one of claims 1-15, **characterized** in that the chemical crosslinkages between the cellulose molecules are formed by one or more substances from the group of crosslinking substances which comprises bifunctional trifunctional or polyfunctional organic acids, bifunctional, trifunctional or polyfunctional aldehydes and also heterocyclic compounds, which have at least two nitrogen atoms in the ring in addition to carbon, including dihydroxyethyleneurea and dimethyldihydroxyethyleneurea and also derivatives of these latter two compounds.

17. Fluff pulp according to claim 16, **characterized** in that the crosslinkages are formed by reactions which are catalyzed by one or more of the substances which comprise the groups alkali metal hypophosphite, alkali metal polyphosphate, alkali metal phosphate, alkali metal sulphate, sodium fluoroborate, disodium carbonate, Lewis acids, such as irontrichloride, and organic amines.
18. Fluffpulp according to any one of claims 1-17, **characterized** in that the cellulose fibres which are subjected to crosslinking are selected from among the groups which comprise bleached, partially bleached and unbleached, sulphate-delignified or sulphite-delignified, softwood and hardwood fibres, thermomechanical and chemothermo-mechanical pulps, and mixtures of the said material, but in that the cellulose fibre preferably consists of bleached softwood sulphate pulp.
19. Fluff pulp according to any one of claims 1-18, **characterized** in that it has the form of a web which is compacted after defibration and crosslinking, and in that it is intended to be defibrated once again in order subsequently to be formed into absorptive pads or into a part of absorptive pads having low density.
20. Absorptive pad, **characterized** in that it consists, at least in part, of the fluff pulp according to any one of claims 1-19.
21. Absorptive pad according to claim 20, **characterized** in that the said fluff pulp exists in a mixture together with superabsorbent polymers (SAP), which are understood to mean polymers which can form gels containing at least 10 g of water/g of polymer.
22. Absorptive pad according to claim 20 or 21, **characterized** in that it also contains synthetic fibres as reinforcing fibres, comprising synthetic fibres, preferably in a mixture together with the said fluff pulp.
23. Absorptive pad according to any one of claims 20-22, **characterized** in that the fluff pulp according to any one of claims 1-19 exists in a mixture together with, or layered together with, cellulose fibres which are not crosslinked.
24. Absorptive pad according to any one of claims 20-23, **characterized** in that it exhibits a density gradient.
25. Absorptive pad according to claim 24, **characterized** in that it is produced from two or more different variants of the fluff pulp according to any one of claims 1-18, which variants have been formed and compressed to different densities before being assembled together.
26. Absorptive pad according to claim 25, **characterized** in that it exhibits layers having different compressibilities and consequently different densities after pressing, which layers can be obtained by adding different relative quantities of the said bifunctional, trifunctional or polyfunctional alcohols or equivalent impregnating agents.
27. Absorptive pad according to any one of claims 20-26, **characterized** in that it consists of one of the products comprising babies' nappies, sanitary towels and incontinence products, or of another product intended to collect body fluids.
28. Absorptive pad according to claims 27, **characterized** in that it exhibits one or more layers intended to face the wearer, which layer(s) has/have (a) lower density (densities) than underlying layers in order to be able to take up relatively large quantities of fluid in a short period of time.
29. Method for preparing fluff pulp, comprising defibrating the cellulose fibres and crosslinking them, **characterized** in that the cellulose fibres are impregnated with an effective quantity of chemicals, comprising crosslinking agent and at least one bifunctional, trifunctional or polyfunctional alcohol, in that the impregnated substance is dried, in that it is defibrated, at the latest after drying, and in that the defibrated cellulose fibres are crosslinked in the dry state at a temperature of between 100 and 210°C as a result of which a fluff pulp is obtained having good compressibility under the influence of heat and pressure in combination with good absorption properties.
30. Method according to claim 30, **characterized** in that the crosslinking reaction is carried out in the dry state at a fibre temperature of between 140 and 190°C.
31. Method according to any one of claims 29-30, **characterized** in that the cellulose fibres are impregnated with an effective quantity of chemicals, which cause the fibres to swell and which thereby soften the cellulose polymers,

as a result of which the compressibility of the fluff pulp under the influence of heat and pressure is improved.

32. Method according to any one of claims 29-31, **characterized** in that the said bifunctional, trifunctional or polyfunctional alcohols have a molecular weight which exceeds 60 g/mole.
33. Method according to any one of claims 29-32, **characterized** in that the carbon structure of the said bifunctional, trifunctional or polyfunctional alcohols contain one or more heteroatoms, which heteroatoms can comprise any one of oxygen or nitrogen.
34. Method according to any one of claims 29-33, **characterized** in that the said bifunctional, trifunctional or polyfunctional alcohols contain one or two polar, functional groups of the type of groups which comprise aldehyde groups, keto groups and carboxyl groups.
35. Method according to any one of claims 29-34, **characterized** in that the cellulose fibres are impregnated with 2-150 g of a bifunctional, trifunctional or polyfunctional alcohol/kg of cellulose fibres.
36. Method according to any one of claims 29-35, **characterized** in that the cellulose fibres are impregnated with 5-50 g of a bifunctional, trifunctional or polyfunctional alcohol/kg of cellulose fibres.
37. Method according to any one of claims 29-36, **characterized** in that the cellulose fibres are impregnated with glycerol.
38. Method according to any one of claims 29-37, **characterized** in that a quantity of crosslinking reagent is added which corresponds to 10-150 g of crosslinking agent/kg of cellulose fibre.
39. Method according to any one of claims 29-38, **characterized** in that a quantity of crosslinking reagent is added which corresponds to 20-60 g of crosslinking agent/kg of cellulose fibre.
40. Method according to any one of claims 29-39, **characterized** in that one or more substances from the group of crosslinking substances which comprises bifunctional, trifunctional or polyfunctional organic acids, bifunctional, trifunctional or polyfunctional aldehydes and also heterocyclic compounds which have at least two nitrogen atoms in the ring in addition to carbon, including dihydroxyethyleneurea and dimethyldihydroxyethyleneurea and derivatives of these two latter compounds, are added as crosslinking reagent.
41. Method according to any one of claims 29-40, **characterized** in that the crosslinking reaction is catalyzed by substances which are selected from one of the groups alkali metal hypophosphite, alkali metal polyphosphate, alkali metal phosphate, alkali metal sulphate, sodium fluoroborate, disodium carbonate, Lewis acids, such as iron trichloride, and organic amines.
42. Method according to any one of claims 29-41, **characterized** in that the cellulose fibres which are subjected to crosslinking are selected from among the groups which comprise bleached, partially bleached and unbleached, sulphate-delignified or sulphite-delignified, softwood and hardwood fibres, thermomechanical and chemothermo-mechanical pulps, and mixtures of the said materials, preferably bleached softwood sulphate pulp.

Patentansprüche

1. Defibrierter, quervernetzte Zellulosefasern enthaltender Flockenzellstoff mit guter Kompressibilität unter Einfluß von Hitze und Druck, dadurch gekennzeichnet, daß der Flockenzellstoff eine Faserstruktur aufweist, die erhältlich ist durch
 - (i) Imprägnieren der Fasern mit einem Quervernetzungs-mittel und wenigstens einem bifunktionalen, trifunktionalen oder polyfunktionalen Alkohol,
 - (ii) Trocknen der Fasern,
 - (iii) Defibrieren des Zellstoffes und
 - (iv) Quervernetzen der Fasern des defibrierten Zellstoffes bei einer Temperatur zwischen 100 und 210°C.
2. Flockenzellstoff nach Anspruch 1, dadurch gekennzeichnet, daß die Zellulosefasern bei einer Temperatur von über

120°C quervernetzt wurden.

3. Flockenzellstoff nach Anspruch 2, dadurch gekennzeichnet, daß die Zellulosefasern bei einer Temperatur zwischen 140 und 190°C quervernetzt wurden.
- 5 4. Flockenzellstoff nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß die quervernetzten Zellulosefasern aus gequollenen und dadurch weichgemachten Zellulosepolymeren bestehen.
- 10 5. Flockenzellstoff nach einem der Ansprüche 1 bis 4, dadurch gekennzeichnet, daß er eine um mindestens 15% höhere Dichte aufweist, nachdem er unter denselben Bedingungen, was Komprimierungszeit, Wärme und Druck betrifft, gepresst worden ist, als ein Zelluloseerzeugnis, dessen Fasern nicht mit einem bifunktionalen, trifunktionalen oder polyfunktionalen Alkohol oder mit einem anderen Präparat, das einen äquivalenten Effekt bewirkt, imprägniert worden sind, wobei die Fasern jedoch vom selben Typ sind und mit demselben Mittel quervernetzt sind als die Fasern in dem zuerst genannten Flockenzellstoff.
- 15 6. Flockenzellstoff nach einem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß die bifunktionalen, trifunktionalen oder polyfunktionalen Alkohole ein Molekulargewicht von mehr als 60 g/Mol aufweisen.
- 20 7. Flockenzellstoff nach einem der Ansprüche 1 bis 6, dadurch gekennzeichnet, daß die Kohlenstoffstruktur des bifunktionalen, trifunktionalen oder polyfunktionalen Alkoholes ein oder mehrere Heteroatome enthält, wobei die Heteroatome Sauerstoff oder Stickstoff umfassen können.
- 25 8. Flockenzellstoff nach einem der Ansprüche 1 bis 7, dadurch gekennzeichnet, daß die bifunktionalen, trifunktionalen oder polyfunktionalen Alkohole ein oder zwei polare funktionale Gruppen des Typs, der Aldehydgruppen, Keto-
gruppen und Carboxylgruppen umfaßt, enthalten.
9. Flockenzellstoff nach einem der Ansprüche 1 bis 8, dadurch gekennzeichnet, daß die Zellulosefasern mit 2-150 g eines bifunktionalen, trifunktionalen oder polyfunktionalen Alkohols/kg Zellulosefasern imprägniert sind.
- 30 10. Flockenzellstoff nach einem der Ansprüche 1 bis 9, dadurch gekennzeichnet, daß die Zellulosefasern mit 5-50 g eines bifunktionalen, trifunktionalen oder polyfunktionalen Alkohols/kg Zellulosefasern imprägniert sind.
- 35 11. Flockenzellstoff nach einem der Ansprüche 1 bis 10, dadurch gekennzeichnet, daß er eine Faserstruktur mit einer verbesserten Kompressibilität unter dem Einfluß von Wärme und Druck aufweist, erhältlich durch Imprägnieren der Zellulosefasern mit einer wirksamen Menge Glycerin.
- 40 12. Flockenzellstoff nach einem der Ansprüche 1 bis 11, dadurch gekennzeichnet, daß, wenn ein aus dem Zelluloseerzeugnis im Luftstrom abgelegtes absorptives Pad zu einer Trockendichte gepreßt wird, die mindestens 50 kg/m³ höher ist als diejenige eines im Luftstrom abgelegten absorptiven Pads, welches aus unbehandelten Fasern hergestellt wurde, es ein spezifisches Feuchtvolumen aufweist, welches mindestens im wesentlichen demjenigen des unbehandelten, verdichteten Erzeugnisses entspricht, d. h. einem Zelluloseerzeugnis im wesentlichen bestehend aus Zellulosefasern, die weder quervernetzt noch mit bifunktionalen, trifunktionalen oder polyfunktionalen Alkoholen oder mit irgendwelchen anderen äquivalenten Reagenzien imprägniert sind.
- 45 13. Flockenzellstoff nach einem der Ansprüche 1 bis 12, dadurch gekennzeichnet, daß, wenn ein aus dem Zelluloseerzeugnis im Luftstrom abgelegtes absorptives Pad zu einer Trockendichte gepreßt wird, die mindestens 50 kg/m³ ist als diejenige eines im Luftstrom abgelegten absorptiven Pads, welches aus unbehandelten Fasern hergestellt wurde, es eine Absorptionskapazität aufweist, die mindestens so groß oder größer ist als die des komprimierten Erzeugnisses, das aus der unbehandelten Zellulosepulpe hergestellt wurde, d. h. einem Zelluloseerzeugnis, das im wesentlichen aus Zellulosefasern besteht, die weder quervernetzt noch mit bifunktionalen, trifunktionalen oder polyfunktionalen Alkoholen oder mit irgendwelchen anderen äquivalenten Reagenzien imprägniert worden sind, wobei das aus unbehandelten Zellulosefasern hergestellte absorptive Pad auf 200 kg/m³ komprimiert wurde.
- 50 14. Flockenzellstoff nach einem der Ansprüche 1 bis 13, dadurch gekennzeichnet, daß er 10-150 g Quervernetzungsmittel/kg Zellulosefasern enthält.
- 55 15. Flockenzellstoff nach einem der Ansprüche 1 bis 14, dadurch gekennzeichnet, daß er 20-60 g Quervernetzungsmittel/kg Zellulosefasern enthält.

mittel/kg Zellulosefasern enthält.

16. Flockenzellstoff nach einem der Ansprüche 1-15, dadurch gekennzeichnet, daß die chemischen Quervernetzungen zwischen den Zellulosemolekülen durch eine oder mehrere Substanzen aus der Gruppe von Quervernetzungs-
5 mitteln gebildet werden, umfassend bifunktionale, trifunktionale oder polyfunktionale organische Säuren, bifunktionale, trifunktionale oder polyfunktionale Aldehyde sowie heterozyklische Verbindungen, die mindestens zwei Stickstoffatome im Ring zusätzlich zu Kohlenstoff aufweisen, einschließlich Dihydroxyethylenharnstoff und Dimethyldihydroxyethylenharnstoff sowie Derivate dieser beiden letzteren Verbindungen.
17. Flockenzellstoff nach Anspruch 16, dadurch gekennzeichnet, daß die Quervernetzungen gebildet werden durch
10 Reaktionen, die durch eine oder mehrere der Substanzen katalysiert werden, die die Gruppen Alkalimetallhypophosphit, Alkalimetallpolyphosphat, Alkalimetallphosphat, Alkalimetallsulfat, Natriumfluoroborat, Dinatriumcarbonat, Lewis-Säuren, wie Eisen(III)-chlorid, und organische Amine umfassen.
18. Flockenzellstoff nach einem der Ansprüche 1 bis 17, dadurch gekennzeichnet, daß die Zellulosefasern, die der
15 Quervernetzung unterzogen werden, ausgewählt sind aus den Gruppen, umfassend gebleichte, teilweise gebleichte und ungebleichte, nach dem Sulfatverfahren delignifizierte oder nach dem Sulfitverfahren delignifizierte Nadelholz- und Laubholzfasern, thermomechanische und chemothermomechanische Holzstoffe und Mischungen aus solchem Material, wobei die Zellulosefasern jedoch vorzugsweise aus gebleichtem Nadelholzsulfatzellstoff bestehen.
20
19. Flockenzellstoff nach einem der Ansprüche 1 bis 18, dadurch gekennzeichnet, daß er die Form eines Faserflores aufweist, der nach Defibrieren und Quervernetzen verdichtet wird, und dadurch, daß er nochmals defibriert werden soll, um nachfolgend zu absorptiven Pads oder zu einem Teil von absorptiven Pads mit geringer Dichte geformt
25 zu werden.
20. Absorptives Pad, dadurch gekennzeichnet, daß es zumindest teilweise aus dem Flockenzellstoff nach einem der Ansprüche 1 bis 19 besteht.
21. Absorptives Pad nach Anspruch 20, dadurch gekennzeichnet, daß der Flockenzellstoff in einem Gemisch zusammen mit superabsorbierenden Polymeren (SAP) vorliegt, worunter man Polymere versteht, die Gele bilden können, die mindestens 10 g Wasser/g Polymer enthalten.
30
22. Absorptives Pad nach Anspruch 20 oder 21, dadurch gekennzeichnet, daß es auch synthetische Fasern als verstärkende Fasern enthält, umfassend synthetische Fasern, vorzugsweise in einem Gemisch zusammen mit dem Flockenzellstoff.
35
23. Absorptives Pad nach einem der Ansprüche 20 bis 22, dadurch gekennzeichnet, daß der Flockenzellstoff nach einem der Ansprüche 1 bis 19 in einem Gemisch oder in Schichten angeordnet zusammen mit Zellulosefasern, die nicht quervernetzt sind, vorliegt.
40
24. Absorptives Pad nach einem der Ansprüche 20 bis 23, dadurch gekennzeichnet, daß es einen Dichtegradienten aufweist.
25. Absorptives Pad nach Anspruch 24, dadurch gekennzeichnet, daß es aus zwei oder mehreren verschiedenen
45 Varianten des Flockenzellstoffs nach einem der Ansprüche 1 bis 18 hergestellt ist, wobei die Varianten geformt und zu unterschiedlichen Dichten gepreßt worden sind, bevor sie zusammen angeordnet wurden.
26. Absorptives Pad nach Anspruch 25, dadurch gekennzeichnet, daß es Schichten mit unterschiedlichen Kompressibilitäten und folglich unterschiedlichen Dichten nach dem Pressen aufweist, wobei die Schichten erhältlich sind durch Zugabe unterschiedlicher relativer Dichten des bifunktionalen, trifunktionalen oder polyfunktionalen Alkohols oder äquivalenter Imprägniermittel.
50
27. Absorptives Pad nach einem der Ansprüche 20 bis 26, dadurch gekennzeichnet, daß es aus einem der Produkte besteht, umfassend Babywindeln, Damenbinden und Inkontinenzprodukte, oder aus einem anderen Produkt, das zum Auffangen von Körperflüssigkeiten dient.
55
28. Absorptives Pad nach Anspruch 27, dadurch gekennzeichnet, daß es eine oder mehrere dem Träger zugewandte

Schichten aufweist, wobei die Schicht(en) eine niedrigere Dichte aufweisen als darunterliegende Schichten, so daß sie in der Lage sind, relativ große Flüssigkeitsmengen in einer kurzen Zeitspanne aufzunehmen.

29. Verfahren zur Herstellung von Flockenzellstoff, umfassend Defibrieren und Quervernetzen der Zellulosefasern, dadurch gekennzeichnet, daß die Zellulosefasern imprägniert sind mit einer wirksamen Menge an Chemikalien, umfassend Quervernetzungsmittel und mindestens einen bifunktionalen, trifunktionalen oder polyfunktionalen Alkohol, daß die imprägnierte Substanz getrocknet wird, daß sie spätestens nach dem Trocknen defibriert wird, und daß die defibrierten Zellulosefasern in trockenem Zustand bei einer Temperatur zwischen 100 und 210°C quervernetzt werden, wodurch ein Flockenzellstoff erhalten wird, der unter dem Einfluß von Hitze und Druck gute Kompressibilität kombiniert mit guten Absorptionseigenschaften aufweist.
30. Verfahren nach Anspruch 29, dadurch gekennzeichnet, daß die Quervernetzungsreaktion im trockenen Zustand bei einer Fasertemperatur zwischen 140 und 190°C durchgeführt wird.
31. Verfahren nach einem der Ansprüche 29 bis 30, dadurch gekennzeichnet, daß die Zellulosefasern mit einer wirksamen Menge an Chemikalien imprägniert werden, die ein Quellen der Fasern bewirken und die dadurch die Zellulosepolymere weichmachen, wodurch die Kompressibilität des Flockenzellstoffs unter dem Einfluß von Wärme und Druck verbessert wird.
32. Verfahren nach einem der Ansprüche 29 bis 31, dadurch gekennzeichnet, daß die bifunktionalen, trifunktionalen oder polyfunktionalen Alkohole ein Molekulargewicht von mehr als 60 g/Mol aufweisen.
33. Verfahren nach einem der Ansprüche 29 bis 32, dadurch gekennzeichnet, daß die Kohlenstoffstruktur der bifunktionalen, trifunktionalen oder polyfunktionalen Alkohole ein oder mehrere Heteroatome enthält, wobei die Heteroatome Sauerstoff oder Stickstoff umfassen können.
34. Verfahren nach einem der Ansprüche 29 bis 33, dadurch gekennzeichnet, daß die bifunktionalen, trifunktionalen oder polyfunktionalen Alkohole ein oder zwei polare funktionale Gruppen des Typs umfassend Aldehydgruppen, Ketogruppen und Carboxylgruppen enthalten.
35. Verfahren nach einem der Ansprüche 29 bis 34, dadurch gekennzeichnet, daß die Zellulosefasern mit 2-150 g eines bifunktionalen, trifunktionalen oder polyfunktionalen Alkohols/kg Zellulosefasern imprägniert werden.
36. Verfahren nach einem der Ansprüche 29 bis 35, dadurch gekennzeichnet, daß die Zellulosefasern mit 5-50 g eines bifunktionalen, trifunktionalen oder polyfunktionalen Alkohols/kg Zellulosefasern imprägniert werden.
37. Verfahren nach einem der Ansprüche 29 bis 36, dadurch gekennzeichnet, daß die Zellulosefasern mit Glycerin imprägniert werden.
38. Verfahren nach einem der Ansprüche 29 bis 37, dadurch gekennzeichnet, daß eine Menge an Quervernetzungsreagens zugegeben wird, die 10-150 g Quervernetzungsmittel/g Zellulosefasern entspricht.
39. Verfahren nach einem der Ansprüche 29 bis 38, dadurch gekennzeichnet, daß eine Menge an Quervernetzungsmittel zugegeben wird, die 20-60 g Quervernetzungsmittel/kg Zellulosefasern entspricht.
40. Verfahren nach einem der Ansprüche 29 bis 39, dadurch gekennzeichnet, daß eine oder mehrere Substanzen aus der Gruppe von Quervernetzungsmitteln, die bifunktionale, trifunktionale oder polyfunktionale organische Säuren, bifunktionale, trifunktionale oder polyfunktionale Aldehyde sowie heterozyklische Verbindungen, die mindestens zwei Stickstoffatome im Ring zusätzlich zu Kohlenstoff aufweisen, einschließlich Dihydroxyethylenharnstoff und Dimethyldihydroxyethylenharnstoff und Derivate dieser zwei letzteren Verbindungen, umfaßt, als Quervernetzungsmittel zugegeben werden.
41. Verfahren nach einem der Ansprüche 29 bis 40, dadurch gekennzeichnet, daß die Quervernetzungsreaktion katalysiert wird durch Substanzen, die ausgewählt sind aus einer der Gruppen Alkalimetallhypophosphit, Alkalimetallpolyphosphat, Alkalimetallphosphat, Alkalimetallsulfat, Natriumfluoroborat, Dinatriumcarbonat, Lewis-Säuren, wie Eisen(III)-chlorid, und organische Amine.
42. Verfahren nach einem der Ansprüche 29 bis 41, dadurch gekennzeichnet, daß die Zellulosefasern, die der Quer-

vernetzung unterzogen werden, ausgewählt sind aus den Gruppen, umfassend gebleichte, teilweise gebleichte und ungebleichte, mit dem Sulfatverfahren delignifizierte oder dem Sulfiterverfahren delignifizierte Nadelholz- und Laubholzfasern, thermomechanische und chemothermomechanische Holzstoffe, und Gemische dieser Materialien, vorzugsweise gebleichten Nadelholzsulfat-Zellstoff.

5

Revendications

1. Mousse de cellulose défibrée contenant des fibres de cellulose réticulées, ayant une bonne compressibilité sous l'influence de la chaleur et de la pression, caractérisée en ce que la mousse de cellulose a une structure des fibres que l'on peut obtenir par (i) imprégnation des fibres avec un agent de réticulation et au moins un alcool bifonctionnel, trifonctionnel ou polyfonctionnel, (ii) séchage des fibres, (iii) défibrage de la pâte, et (iv) réticulation des fibres de la pâte défibrée à une température entre 100 et 210°C.
2. Mousse de cellulose selon la revendication 1, caractérisée en ce que les fibres de cellulose ont été réticulées à une température supérieure à 120°C.
3. Mousse de cellulose selon la revendication 2, caractérisée en ce que les fibres de cellulose ont été réticulées à une température entre 140 et 190°C.
4. Mousse de cellulose selon l'une quelconque des revendications 1 à 3, caractérisée en ce que les fibres de cellulose réticulées se composent de polymères de cellulose gonflés, et de ce fait ramollis.
5. Mousse de cellulose selon l'une quelconque des revendications 1 à 4, caractérisée en ce que, une fois comprimée, elle a une densité qui est supérieure d'au moins 15%, dans les mêmes conditions en ce qui concerne la durée de compression, la chaleur et la pression, à celle d'un produit de cellulose dont les fibres n'ont pas été imprégnées d'un alcool bifonctionnel, trifonctionnel ou polyfonctionnel, ou de quelqu'autre préparation qui fournit un effet équivalent, mais lesquelles fibres sont sans cela du même type, et sont réticulées avec le même agent, que les fibres de la mousse de cellulose nommée en premier.
6. Mousse de cellulose selon l'une quelconque des revendications 1 à 5, caractérisée en ce que lesdits alcools bifonctionnels, trifonctionnels ou polyfonctionnels ont un poids moléculaire qui excède 60g/mole.
7. Mousse de cellulose selon l'une quelconque des revendications 1 à 6, caractérisée en ce que la structure de carbone desdits alcools bifonctionnels, trifonctionnels ou polyfonctionnels contient un ou plusieurs hétéroatomes, lesquels hétéroatomes peuvent comprendre n'importe lequel de l'oxygène et de l'azote.
8. Mousse de cellulose selon l'une quelconque des revendications 1 à 7, caractérisée en ce que lesdits alcools bifonctionnels, trifonctionnels ou polyfonctionnels contiennent un ou deux groupes fonctionnels polaires du type de groupes qui comprennent des groupes aldéhydes, des groupes céto et des groupes carboxyles.
9. Mousse de cellulose selon l'une quelconque des revendications 1 à 8, caractérisée en ce que les fibres de cellulose sont imprégnées de 2 à 150 g d'un alcool bifonctionnel, trifonctionnel ou polyfonctionnel/kg de fibres de cellulose.
10. Mousse de cellulose selon l'une quelconque des revendications 1 à 9, caractérisée en ce que les fibres de cellulose sont imprégnées de 5 à 50 g d'un alcool bifonctionnel, trifonctionnel ou polyfonctionnel/kg de fibres de cellulose.
11. Mousse de cellulose selon l'une quelconque des revendications 1 à 10, caractérisée en ce qu'elle possède une structure des fibres ayant une compressibilité accrue sous l'influence de la chaleur et de la pression, que l'on peut obtenir par imprégnation de la fibre de cellulose avec une quantité effective de glycérol.
12. Mousse de cellulose selon l'une quelconque des revendications 1 à 11, caractérisée en ce que, lorsqu'un tampon absorbant formé par voie sèche (air-laid) fabriqué à partir du produit de cellulose est comprimé à une densité à sec qui est supérieure d'au moins 50 kg/m³ à celle d'un tampon absorbant formé par voie sèche fabriqué à partir de fibres non traitées, elle a un volume humide spécifique qui correspond au moins essentiellement à celui du produit comprimé et non traité, c'est-à-dire un produit de cellulose essentiellement constitué par des fibres de cellulose qui n'ont été ni réticulées ni imprégnées d'alcools bifonctionnels, trifonctionnels ou polyfonctionnels ou de tout autre réactif équivalent.

13. Mousse de cellulose selon l'une quelconque des revendications 1 à 12, caractérisée en ce que, lorsqu'un tampon absorbant formé par voie sèche fabriqué à partir du produit de cellulose est comprimé à une densité à sec qui est supérieure d'au moins 50 kg/m³ à celle d'un tampon absorbant à couche d'air fabriqué à partir de fibres non traitées, elle a une capacité d'absorption qui est au moins aussi importante ou supérieure à celle du produit comprimé fabriqué à partir de la pâte de cellulose non traitée, c'est-à-dire un produit de cellulose essentiellement constitué par des fibres de cellulose qui n'ont été ni réticulées ni imprégnées d'alcools bifonctionnels, trifonctionnels ou polyfonctionnels ou de tout autre réactif équivalent, lequel tampon absorbant fabriqué à partir de fibres de cellulose non traitées a été comprimé à 200 kg/m³.
14. Mousse de cellulose selon l'une quelconque des revendications 1 à 13, caractérisée en ce qu'elle contient 10 à 150 g d'agent de réticulation/kg de fibres de cellulose.
15. Mousse de cellulose selon l'une quelconque des revendications 1 à 14, caractérisée en ce qu'elle contient 20 à 60 g d'agent de réticulation/kg de fibres de cellulose.
16. Mousse de cellulose selon l'une quelconque des revendications 1 à 15, caractérisée en ce que les liaisons de réticulation chimiques entre les molécules de cellulose sont formées par une ou plusieurs substances du groupe de substances de réticulation qui comporte des acides organiques bifonctionnels, trifonctionnels ou polyfonctionnels, des aldéhydes bifonctionnels, trifonctionnels ou polyfonctionnels et également des composés hétérocycliques, qui ont au moins deux atomes d'azote dans le cycle en plus du carbone, comprenant la dihydroxyéthylèneurée et la diméthylidihydroxyéthylèneurée et également des dérivés de ces deux derniers composés.
17. Mousse de cellulose selon la revendication 16, caractérisée en ce que les liaisons de réticulation sont formées par des réactions qui sont catalysées par une ou plusieurs des substances qui comprennent les groupes hypophosphite de métal alcalin, polyphosphate de métal alcalin, phosphate de métal alcalin, sulfate de métal alcalin, fluoroborate de sodium, carbonate de disodium, acides de Lewis, tels que le trichlorure de fer, et amines organiques.
18. Mousse de cellulose selon l'une quelconque des revendications 1 à 17, caractérisée en ce que les fibres de cellulose qui sont soumises à la réticulation sont choisies dans les groupes qui comprennent des fibres de bois résineux et des fibres de bois durs, blanchies, partiellement blanchies et non blanchies, délignifiées au sulfate ou délignifiées au sulfite, des pâtes thermomécaniques et des pâtes chimiothermomécaniques, et des mélanges dudit matériau, mais en ce que la fibre de cellulose se compose de préférence de pâte blanchie au sulfate de bois résineux.
19. Mousse de cellulose selon l'une quelconque des revendications 1 à 18, caractérisée en ce qu'elle a la forme d'une bande qui est compactée après défibrage et réticulation, et en ce qu'elle est destinée à être défibrée encore une fois afin d'être formée ultérieurement en tampons absorbants ou en une partie de tampons absorbants ayant une faible densité.
20. Tampon absorbant, caractérisé en ce qu'il se compose, au moins en partie, de la mousse de cellulose selon l'une quelconque des revendications 1 à 19.
21. Tampon absorbant selon la revendication 20, caractérisé en ce que ladite mousse de cellulose existe dans un mélange avec des polymères superabsorbants (SAP), qui sont présumés signifier des polymères qui peuvent former des gels contenant au moins 10 g d'eau/g de polymère.
22. Tampon absorbant selon la revendication 20 ou 21, caractérisé en ce qu'il contient également des fibres synthétiques comme fibres de renforcement, comprenant des fibres synthétiques, de préférence dans un mélange avec ladite Mousse de cellulose.
23. Tampon absorbant selon l'une quelconque des revendications 20 à 22, caractérisé en ce que la mousse de cellulose selon l'une quelconque des revendications 1 à 19 existe dans un mélange avec, ou disposée en couches avec, des fibres de cellulose qui ne sont pas réticulées.
24. Tampon absorbant selon l'une quelconque des revendications 20 à 23, caractérisé en ce qu'il présente un gradient de densité.

25. Tampon absorbant selon la revendication 24, caractérisé en ce qu'il est fabriqué à partir de deux ou plusieurs variantes différentes de la mousse de cellulose selon l'une quelconque des revendications 1 à 18, lesquelles variantes ont été formées et comprimées à différentes densités avant d'être assemblées les unes avec les autres.
- 5 26. Tampon absorbant selon la revendication 25, caractérisé en ce qu'il présente des couches ayant différentes compressibilités et par conséquent différentes densités après compression, lesquelles couches peuvent être obtenues par addition de différentes quantités relatives desdits alcools bifonctionnels, trifonctionnels ou polyfonctionnels ou d'agents d'imprégnation équivalents.
- 10 27. Tampon absorbant selon l'une quelconque des revendications 20 à 26, caractérisé en ce qu'il consiste en un des produits comprenant les couches de bébé, les serviettes hygiéniques et les produits pour l'incontinence, ou en un autre produit destiné à recueillir les liquides de l'organisme.
- 15 28. Tampon absorbant selon la revendication 27, caractérisé en ce qu'il présente une ou plusieurs couches destinées à affronter le porteur, laquelle(lesquelles) couche(s) a(ont) une(des) densité(s) inférieure(s) aux couches sous-jacentes afin d'être capable(s) d'absorber des quantités relativement importantes de liquide en un court laps de temps.
- 20 29. Procédé de préparation de mousse de cellulose, comportant le défilage des fibres de cellulose et leur réticulation, caractérisé en ce que les fibres de cellulose sont imprégnées d'une quantité effective de produits chimiques, comprenant l'agent de réticulation et au moins un alcool bifonctionnel, trifonctionnel ou polyfonctionnel, en ce que la substance imprégnée est séchée, en ce qu'elle est défibrée, au plus tard après le séchage, et en ce que les fibres de cellulose défibrées sont réticulées à l'état sec à une température entre 100 et 210°C par suite de quoi une mousse de cellulose est obtenue ayant une bonne compressibilité sous l'influence de la chaleur et de la pression combinée à de bonnes propriétés d'absorption.
- 25 30. Procédé selon la revendication 30, caractérisé en ce que la réaction de réticulation est effectuée à l'état sec à une température des fibres entre 140 et 190°C.
- 30 31. Procédé selon l'une quelconque des revendications 29 et 30, caractérisé en ce que les fibres de cellulose sont imprégnées d'une quantité effective de produits chimiques, qui entraînent le gonflement des fibres et qui de ce fait ramollissent les polymères de cellulose, par suite de quoi la compressibilité de la mousse de cellulose sous l'influence de la chaleur et de la pression est accrue.
- 35 32. Procédé selon l'une quelconque des revendications 29 à 31, caractérisé en ce que lesdits alcools bifonctionnels, trifonctionnels ou polyfonctionnels ont un poids moléculaire qui excède 60 g/mole.
33. Procédé selon l'une quelconque des revendications 29 à 32, caractérisé en ce que la structure de carbone desdits alcools bifonctionnels, trifonctionnels ou polyfonctionnels contient un ou plusieurs hétéroatomes, lesquels hétéroatomes peuvent comprendre n'importe lequel de l'oxygène et de l'azote.
- 40 34. Procédé selon l'une quelconque des revendications 29 à 33, caractérisé en ce que lesdits alcools bifonctionnels, trifonctionnels ou polyfonctionnels contiennent un ou deux groupes fonctionnels polaires du type de groupes qui comprennent des groupes aldéhydes, des groupes céto et des groupes carboxyles.
- 45 35. Procédé selon l'une quelconque des revendications 29 à 34, caractérisé en ce que les fibres de cellulose sont imprégnées de 2 à 150 g d'un alcool bifonctionnel, trifonctionnel ou polyfonctionnel/kg de fibres de cellulose.
- 50 36. Procédé selon l'une quelconque des revendications 29 à 35, caractérisé en ce que les fibres de cellulose sont imprégnées de 5 à 50 g d'un alcool bifonctionnel, trifonctionnel ou polyfonctionnel/kg de fibres de cellulose.
37. Procédé selon l'une quelconque des revendications 29 à 36, caractérisé en ce que les fibres de cellulose sont imprégnées de glycérol.
- 55 38. Procédé selon l'une quelconque des revendications 29 à 37, caractérisé en ce qu'une quantité d'agent de réticulation est ajoutée qui correspond à 10 à 150 g d'agent de réticulation/kg de fibre de cellulose.
39. Procédé selon l'une quelconque des revendications 29 à 38, caractérisé en ce qu'une quantité d'agent de réticu-

lation est ajoutée qui correspond à 20 à 60 g d'agent de réticulation/kg de fibre de cellulose.

- 5 40. Procédé selon l'une quelconque des revendications 29 à 39, caractérisé en ce qu'une ou plusieurs substances du groupe de substances de réticulation qui comporte des acides organiques bifonctionnels, trifonctionnels ou polyfonctionnels, des aldéhydes bifonctionnels, trifonctionnels ou polyfonctionnels et également des composés hétérocycliques qui ont au moins deux atomes d'azote dans le cycle en plus du carbone, comprenant la dihydroxyéthylèneurée et la diméthylidihydroxyéthylèneurée et des dérivés de ces deux derniers composés, sont ajoutées comme agent de réticulation.
- 10 41. Procédé selon l'une quelconque des revendications 29 à 40, caractérisé en ce que la réaction de réticulation est catalysée par des substances qui sont choisies dans un des groupes hypophosphite de métal alcalin, polyphosphate de métal alcalin, phosphate de métal alcalin, sulfate de métal alcalin, fluoroborate de sodium, carbonate de disodium, acides de Lewis, tels que le trichlorure de fer, et amines organiques.
- 15 42. Procédé selon l'une quelconque des revendications 29 à 41, caractérisé en ce que les fibres de cellulose qui sont soumises à la réticulation sont choisies dans les groupes qui comprennent des fibres de bois résineux et des fibres de bois durs, blanchies, partiellement blanchies et non blanchies, délignifiées au sulfate ou délignifiées au sulfite, des pâtes thermomécaniques et des pâtes chimiothermomécaniques, et des mélanges desdits matériaux, de préférence la pâte blanchie au sulfate de bois résineux.

Fig.1.

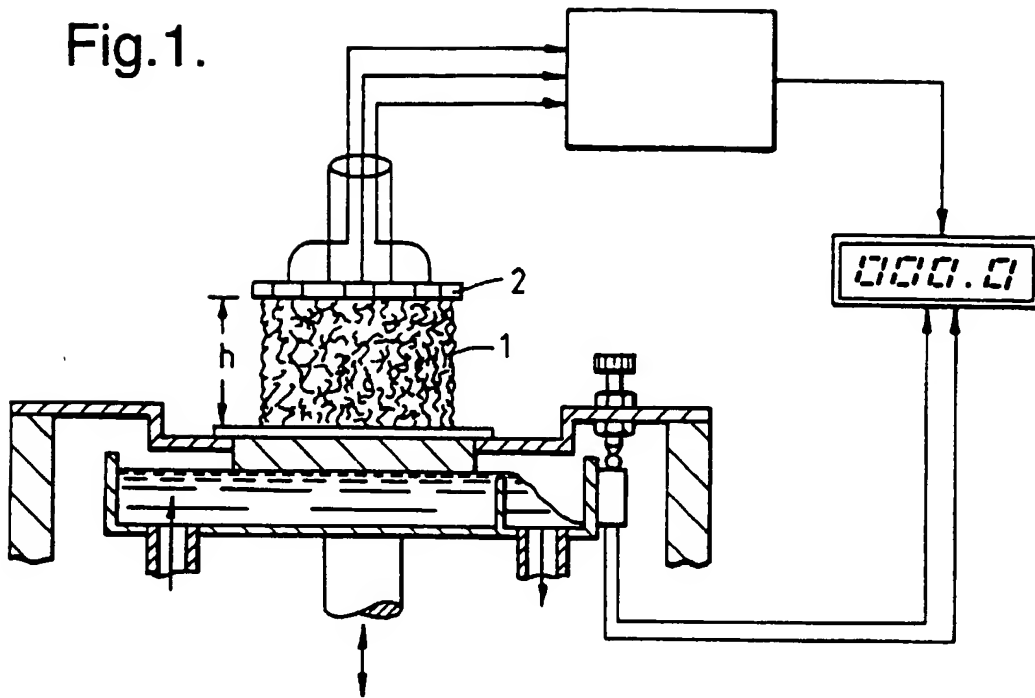


Fig.2.

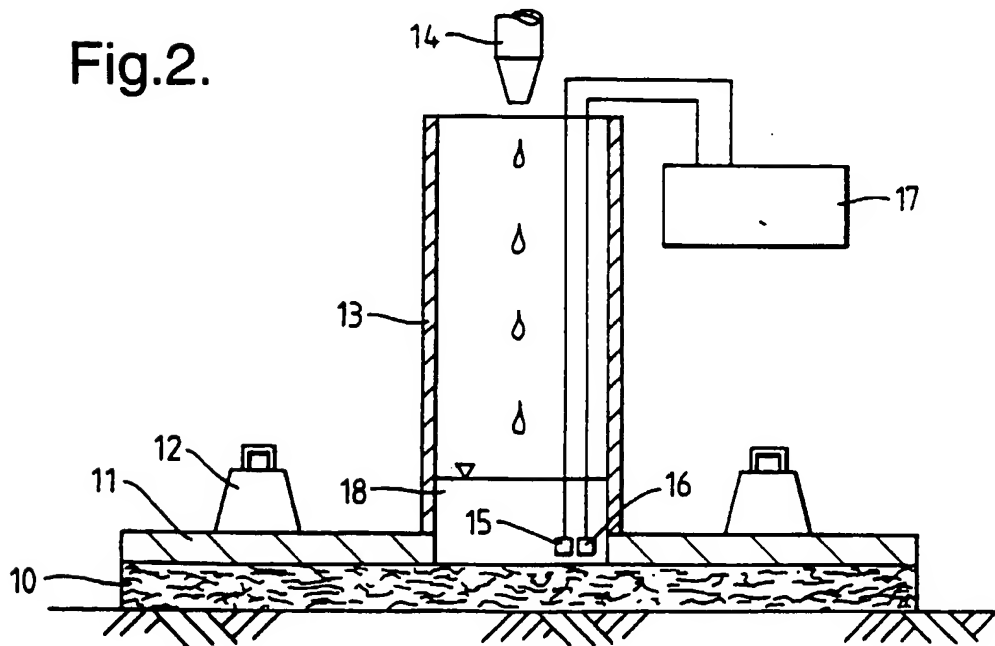


Fig.3.

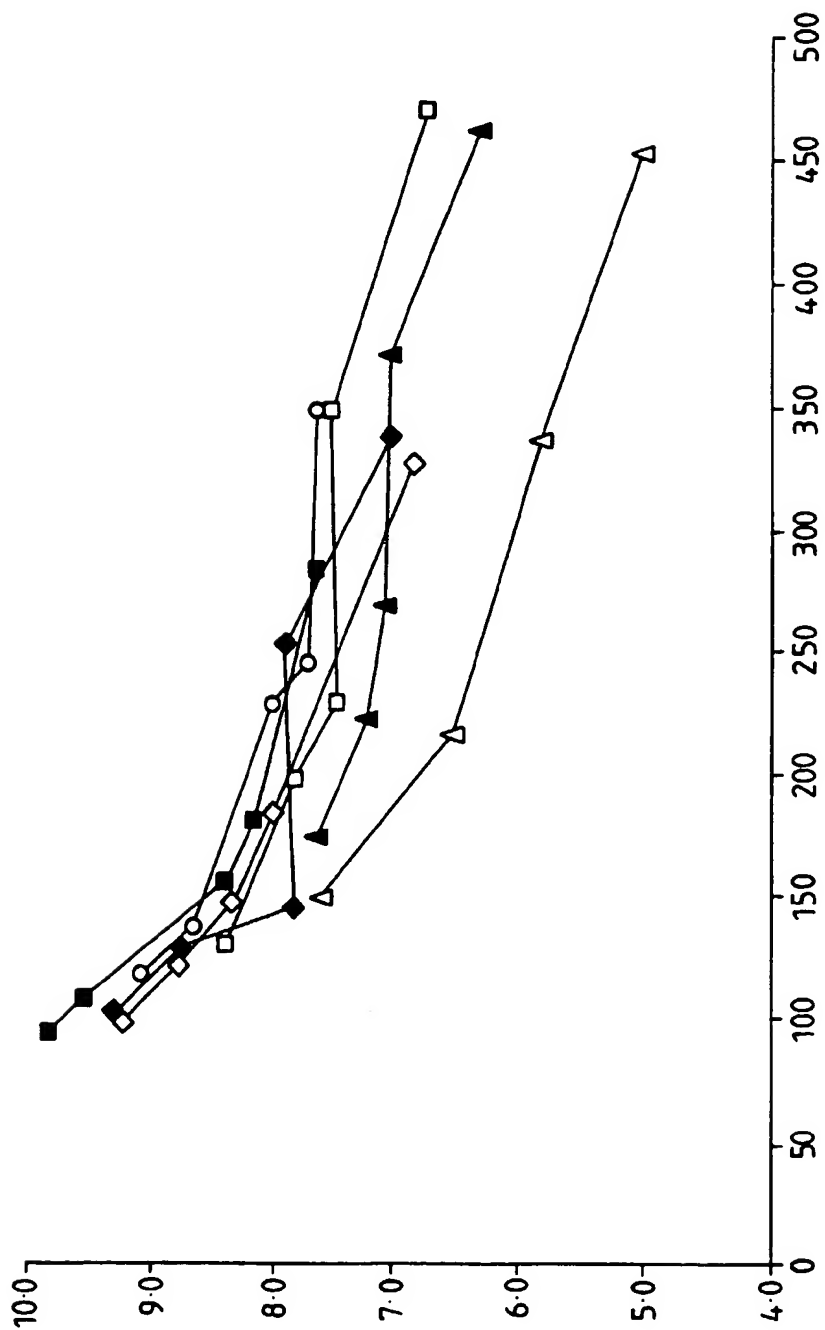


Fig.4.

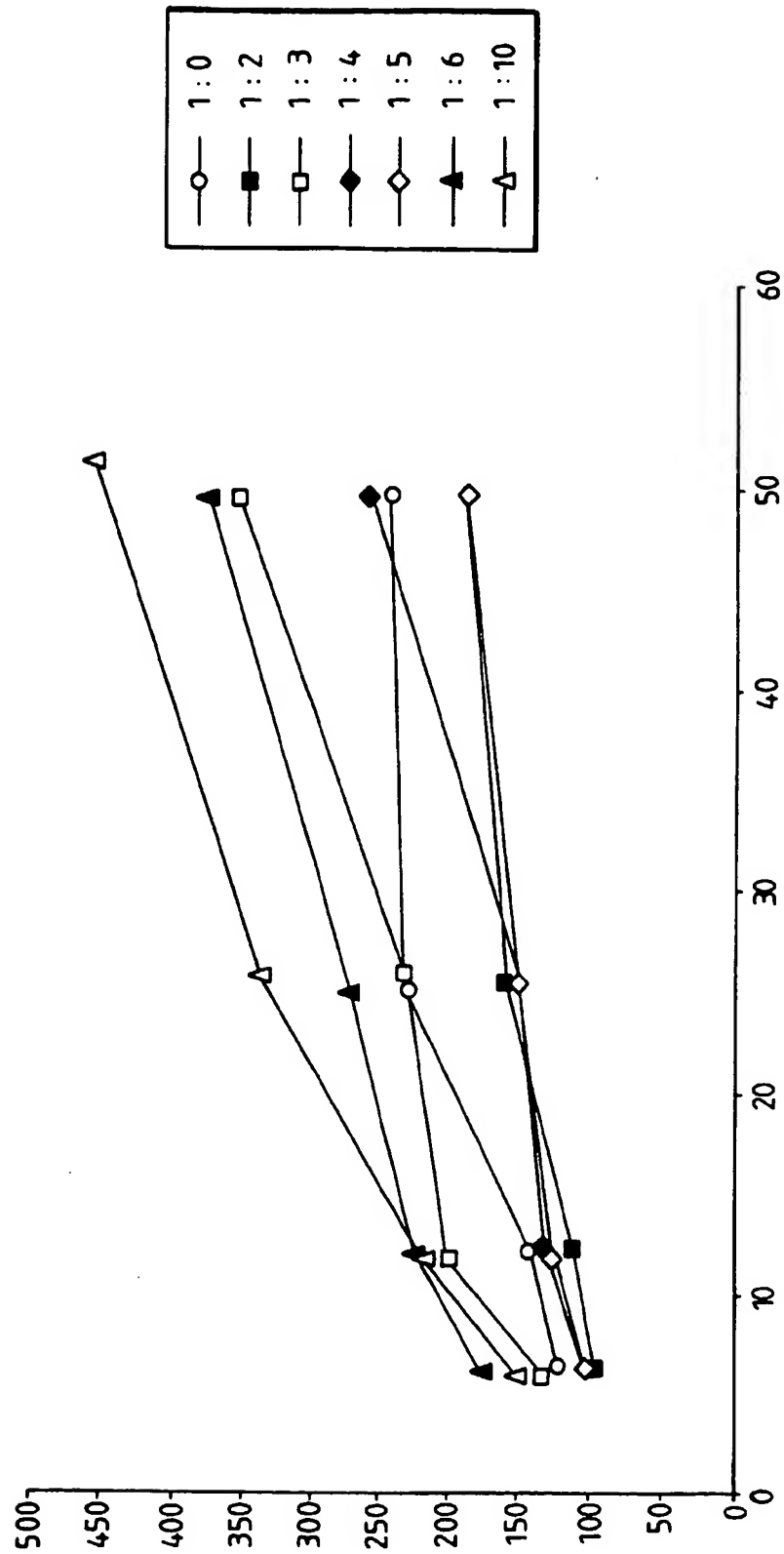


Fig.5.

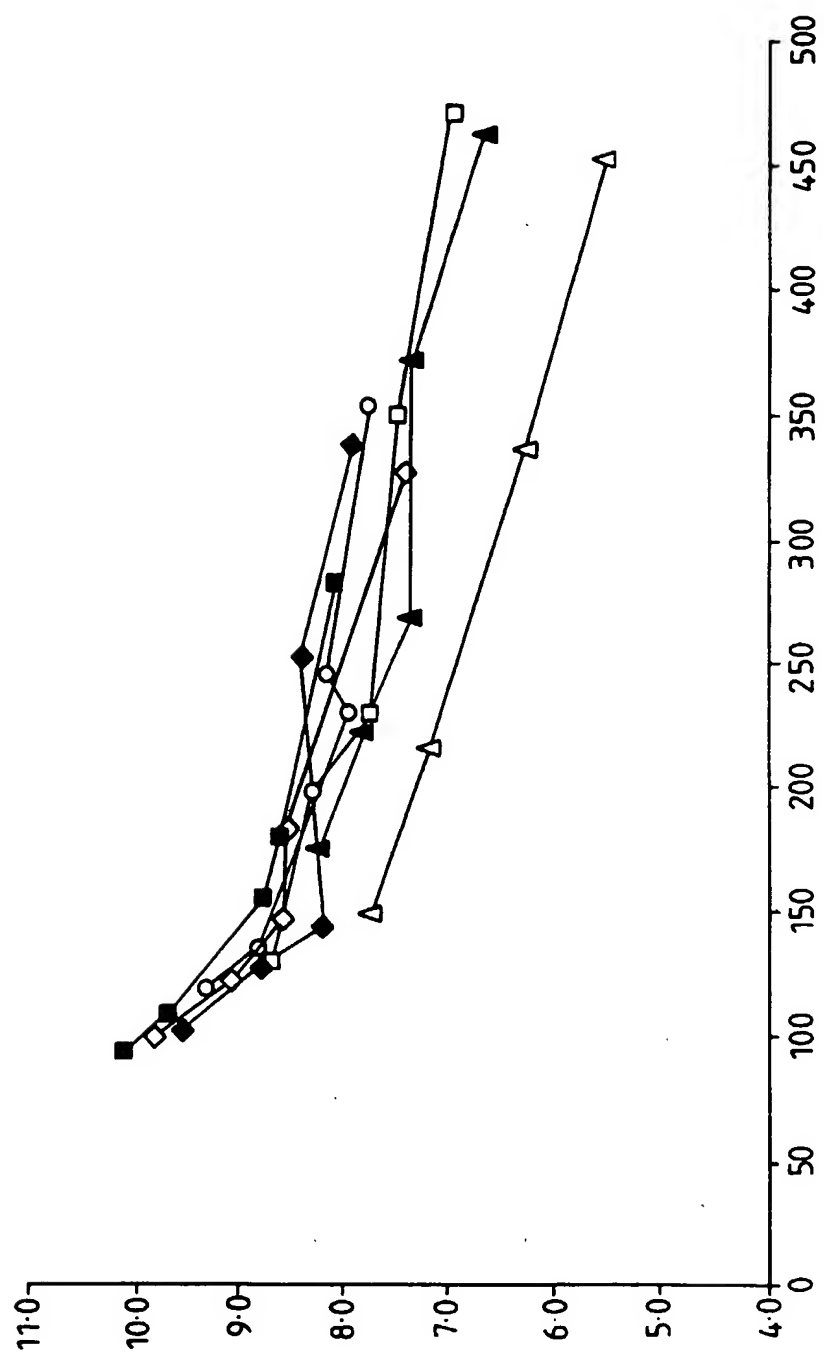


Fig.6.

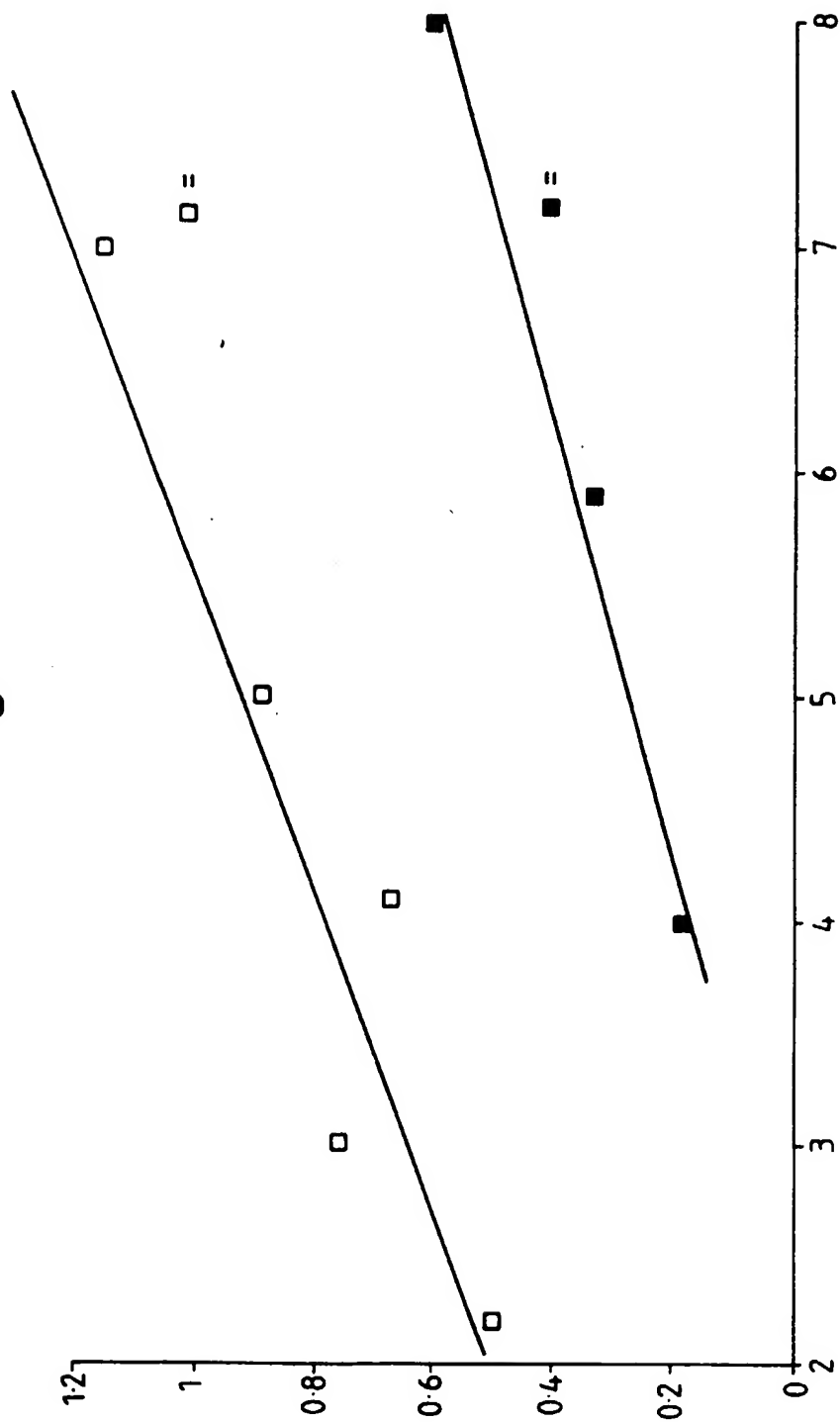


Fig.7.

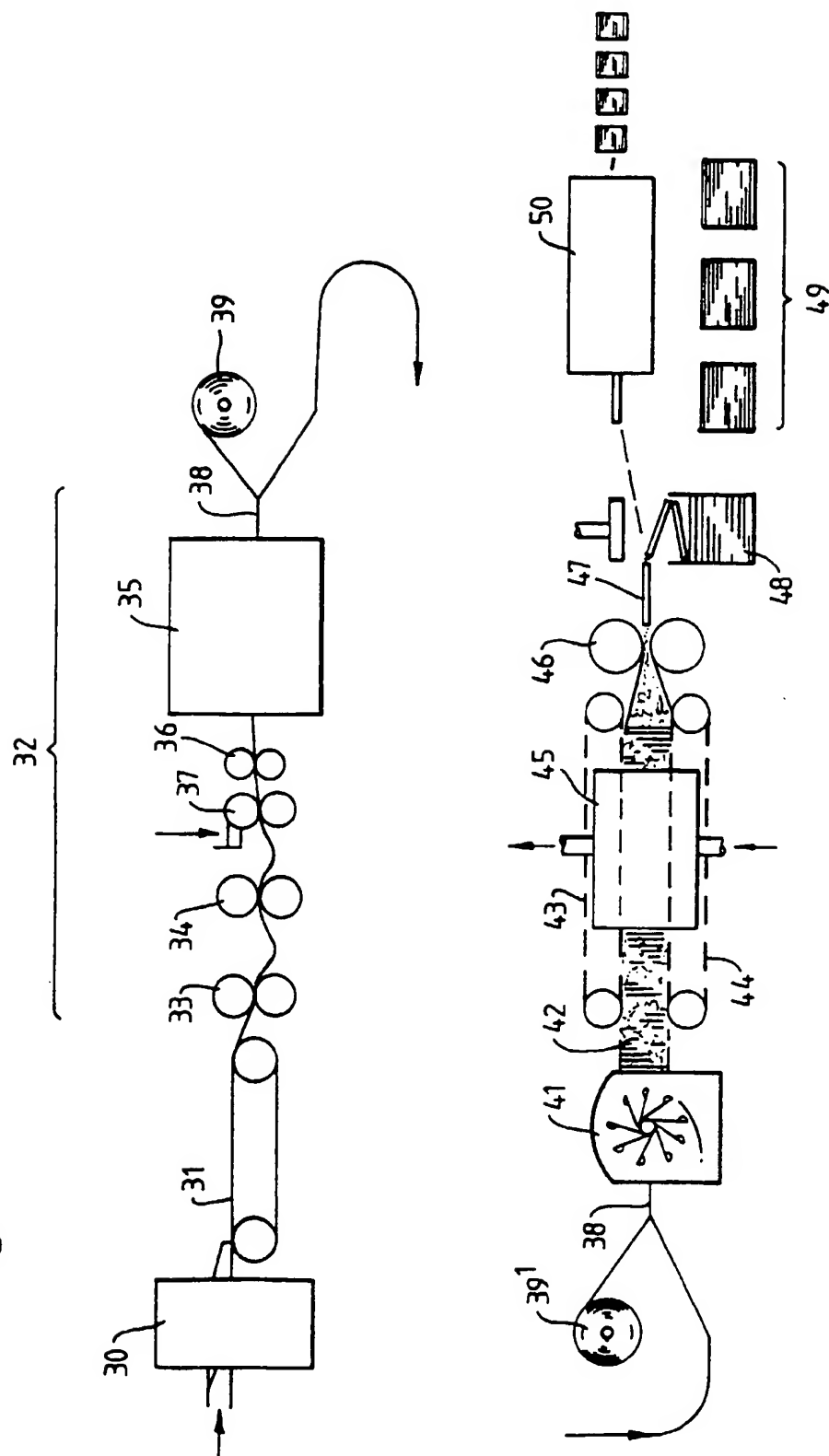


Fig.8.

